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A MICROSCOPIC STUDY OF GOETHITE AND HEMATITE IN THE BROWN IRON ORES OF EAST TEXAS*

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INTRODUCTION

During 1934 Eckel studied the brown iron ores of east Texas for the U. S. Geological Survey.¹ Field relations showed that the ores were formed by the weathering of a greensand. At Mr. Eckel's suggestion, the writer made a study of fifteen polished sections of typical brown ore. The results indicate the value of a line of investigation which has not been extensively applied to the brown iron ores.

The writer is deeply grateful to M. N. Short of the University of Arizona, C. F. Park, Jr., W. T. Schaller, E. F. Burchard and R. C. Wells of the U. S. Geological Survey, and George Tunell of the Geophysical Laboratory for valuable criticism, and especially to E. B. Eckel of the U. S. Geological Survey, who not only furnished the material for this study and helped to direct the progress of the investigation, but also generously contributed the section on geologic relations.

GEOLOGIC RELATIONS²

The brown iron ore deposits of east Texas occupy part of the Coastal Plain Province in the eastern and northeastern parts of the State. All the ores considered here came from the northern part of the field, in Cass, Marion and Morris Counties. The iron ores occur near the tops of the flat-topped, sand-covered hills that are a prominent feature of the landscape in a large part of eastern Texas.

Nearly all the rocks exposed at the surface in this part of the State were laid down during the Eocene division of Tertiary time. Iron ore of commercial grade is confined almost entirely to the Weches greensand member of the Mount Selman formation, which comprises the lower part

* Published by permission of the Director, U. S. Geological Survey.

¹ Eckel, E. B., and Purcell, P. E. M., Iron ores of east Texas: *Univ. of Texas, Bull.* 3401, pp. 485-503, 1935.

² Eckel, E. B., personal communication.

of the Claiborne group. The Weches greensand is a mixture of a granular iron silicate mineral of the glauconite group with varying proportions of quartz, sand, and clay.

The most abundant type of ore is "limonite," or brown ore. In the northern part of the field the ore occurs chiefly in concretionary forms, or as thin lenticular bodies that are distributed irregularly through the weathered zone in the upper part of the Weches greensand. The best ores occur near the outcrop of the Weches and seldom extend beneath heavy cover. Iron carbonate, or siderite, is plentiful in many places. It occurs as white or gray dense nodules or thin lenses, at or near the ground-water level.

Chemical considerations, based on analyses of the greensand, siderite, brown ore, and spring waters, and the observed relation of the ore deposits to the present water table and topography, indicate that the ores have been derived from the greensand by ordinary weathering processes. It is believed that ground waters leach iron from the greensand and deposit it as iron carbonate. This is later altered to "limonite," or brown ore.

THE HYDROUS IRON OXIDES—GOETHITE AND LEPIDOCROCITE

Considerable confusion still exists with regard to the hydrous iron oxides, particularly in the use of the term "limonite." Published results of investigations^{3,4,5,6} carried out during the past few years show that there are two polymorphic forms of ferric oxide monohydrate ($\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$), goethite and lepidocrocite, and that the use of the term "limonite" should be confined to mixtures of ferric iron minerals whose constituents have not been identified, including principally hematite, goethite, lepidocrocite, and jarosite.

IDENTIFICATION AND DESCRIPTION OF THE ORE MINERALS

Hematite, goethite, and lepidocrocite may be identified either by the x-ray powder diffraction method or by measurement of their refractive indices by the immersion method.⁷ The measurement of refractive indices requires the use of high-index melts of sulphur, iodine, selenium, and tellurium which were not available. Identification was therefore made by

³ Posnjak, E., and Merwin, H. E., The hydrated ferric oxides: *Am. Jour. Sci.*, vol. 47, p. 311, 1919.

⁴ The system $\text{Fe}_2\text{O}_3\text{—SO}_3\text{—H}_2\text{O}$, *Jour. Am. Chem. Soc.*, vol. 44, p. 1971, 1922.

⁵ Tunell, G., and Posnjak, E., The stability relationships of goethite and hematite: *Econ. Geol.*, vol. 26, p. 337, 1931.

⁶ Schneiderhöhn, H., and Ramdohr, P., *Lehrbuch der Erzmikroskopie*, Berlin, vol. 2, pp. 560–566, 1931.

⁷ Tunell, G., and Posnjak, E., *op. cit.*, pp. 342–343.

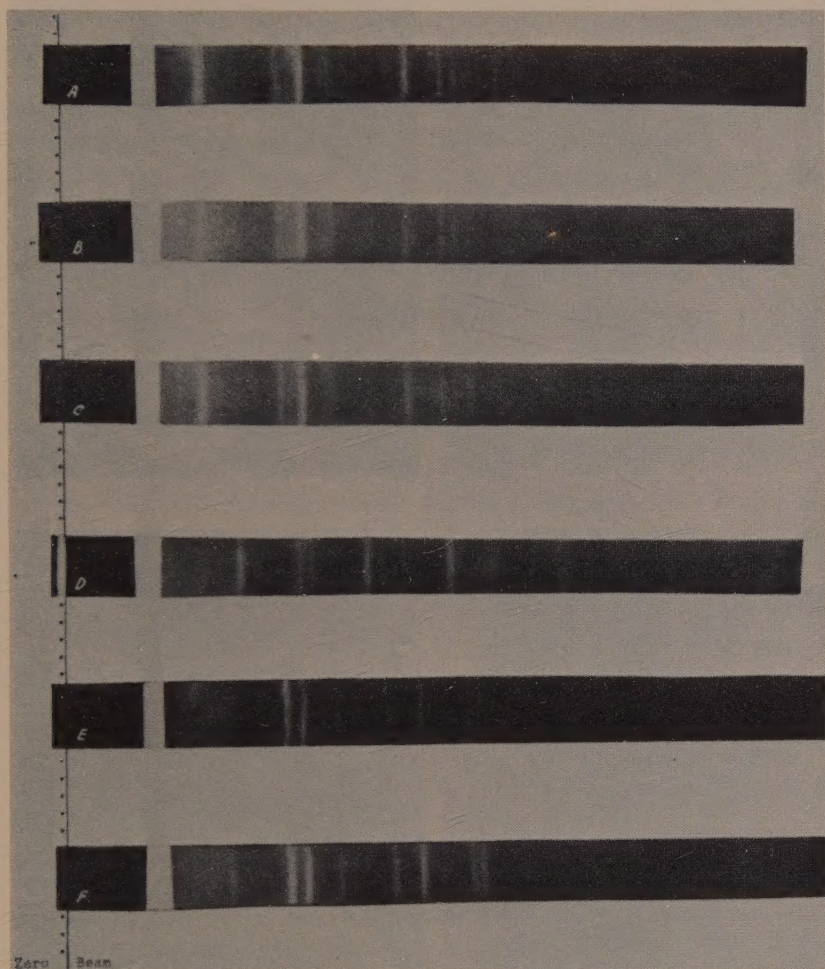


FIG. 1

X-RAY DIFFRACTION PATTERNS

- A. Goethite. Las Vegas, Nevada. U. S. National Museum.
- B. Goethite. Crystalline material from zone 1.
- C. Goethite. Cryptocrystalline material from zones 2 and 3.
- D. Lepidocrocite. Nassau, Germany. U. S. National Museum.
- E. Hematite. Frizzington, England. U. S. National Museum.
- F. Hematite. Material filling vugs in goethite.

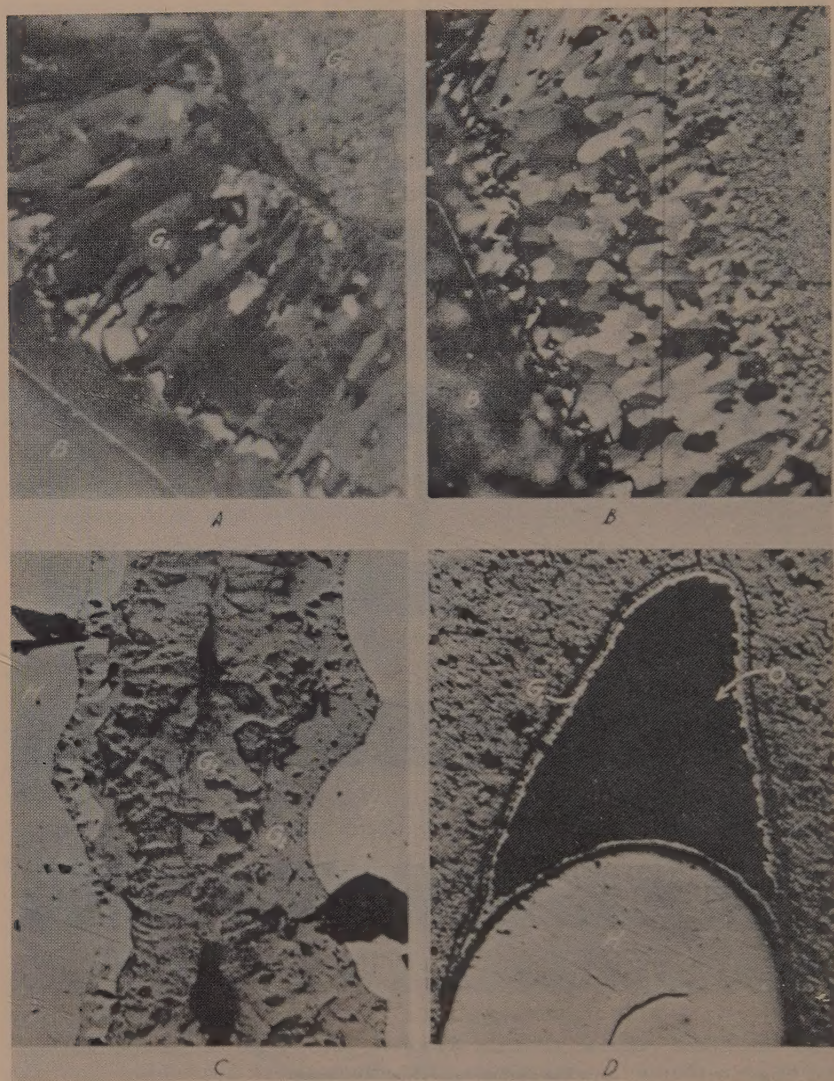


FIG. 2

A. Flamboyant internal structure of a colloform shell of crystalline goethite lining a balsam filled vug in cryptocrystalline goethite. Crossed nicols. Enlarged 68 diameters.

B. Granular internal structure of a part of the same colloform shell of goethite as in A. Crossed nicols. Enlarged 68 diameters.

C. Lobate boundary separating a colloform shell of crystalline hematite from goethite. Plain reflected light. Enlarged 52 diameters.

D. A vug in cryptocrystalline goethite partly filled with crystalline hematite. The unfilled part of the vug is lined with a thin layer of crystalline goethite. Plain reflected light. The narrow black band separating the goethite G_1 from G_2 and hematite from goethite is open space. Enlarged 53 diameters.

G_1 —Goethite (crystalline)

B—Balsam

G_2 —Goethite (cryptocrystalline)

O—Open Space

H—Hematite

comparing x-ray diffraction patterns of the unknown materials with patterns of tested minerals supplied by the U. S. National Museum. The comparison shows that goethite and hematite are present in the east Texas ores and that lepidocrocite is absent. In Fig. 1, the diffraction patterns of B and C, representing crystalline and cryptocrystalline material from the East Texas ores, are identical with that of known goethite from Las Vegas, Nevada (A), and quite different from the pattern obtained from known lepidocrocite from Nassau, Germany (D). The x-ray diffraction patterns were made by R. G. Picard of the Department of Physics, University of Arizona.

The polished sections examined consist of approximately 80 per cent goethite and 20 per cent hematite. The characteristics of the goethite are identical with those described by Schneiderhöhn and Ramdohr.⁸ In vertically reflected light the color varies from dull gray to bright gray. The variation depends largely upon texture, but somewhat upon the orientation of individual grains, as the mineral is faintly pleochroic, and many pure masses of goethite appear to be made up of more than one mineral. Crystalline goethite has a high degree of reflectivity and is brighter than cryptocrystalline material.

The crystalline goethite is strongly anisotropic under crossed nicols, in vertically reflected light, and a delicately flamboyant to coarsely granular structure is brought out (Fig. 2, A and B). The polarization colors range from blue-gray to salmon-brown when the specimen is illuminated by the carbon arc. Owing to the fact that the crystallographic axes could not be identified in polished section it is not possible to designate the corresponding polarization colors. Anisotropism of cryptocrystalline goethite is masked by the yellowish internal reflection. The color of the powdered mineral under crossed nicols in reflected light is brilliant orange-yellow to golden-yellow.

The goethite is stained by a saturated solution of stannous chloride in 1:5 HCl. The stain, which is dark brown on crystalline goethite and yellowish brown on cryptocrystalline material, usually becomes visible in less than one-half minute, but some specimens require treatment for two minutes before even a faint stain appears.

⁸ Schneiderhöhn, H., and Ramdohr, P., *op. cit.*, pp. 561–564.

The hematite in the polished sections of the east Texas ores is easily recognized by its brighter white color and greater hardness than goethite (Fig. 2, C and D). It is strongly anisotropic under crossed nicols in vertically reflected light, with blue-gray to salmon-brown polarization colors and a prominent flamboyant internal structure. The powdered mineral is bright red under crossed nicols in vertically reflected light.

Although *x*-ray diffraction patterns showed lepidocrocite to be absent in the east Texas ores, a polished section of lepidocrocite was studied and the characteristics of the mineral were carefully compared with those of goethite. The differences consist of a somewhat brighter color in plain reflected light and a reddish color of most of the powdered mineral under crossed nicols. The writer believes that these slight differences are insufficient to establish the presence or absence of lepidocrocite without the confirmatory evidence of *x*-ray diffraction patterns or refractive indices.

STRUCTURE OF THE ORES

Roughly ellipsoidal structures characteristic of the iron carbonate concretions from which the brown iron ores have been derived⁹ appear in most of the polished sections. Three zones can be distinguished in most specimens, each of which contains goethite of a different character. Hematite occurs in variable amounts in all three zones.

Zone 1, a central or inner zone, consists mainly of crystalline goethite as a band of varying thickness around a vug, or as a solid core. It takes a relatively high polish and usually is bright gray, although variation in reflectivity is common. In places the grain boundaries are visible in plain vertically reflected light (Fig. 3, C), but under crossed nicols the structure of the goethite is predominantly flamboyant (Fig. 2, A), although some areas are granular (Fig. 2, B). Crushed fragments of the mineral appear flamboyant when immersed in oil in plain transmitted light; under crossed nicols the individual fibers are distinctly anisotropic and have parallel extinctions.

Zone 2 is a concentric envelope of goethite with lower reflectivity and a darker gray color than that of zone 1, and is separated from it by a lobate boundary in most of the sections examined. Under crossed nicols the material is cryptocrystalline and distinctly brown, apparently due to internal reflection. Aggregate anisotropism is weak. Powdered goethite from this zone immersed in oil under crossed nicols does not appear flamboyant, but individual grains are moderately anisotropic.

The goethite in the outer zone 3 is soft and porous, with a much lower reflectivity and duller color than that of zone 2. Under crossed nicols

⁹ Eckel, E. B., and Purcell, P. E. M., *op. cit.*, p. 494.

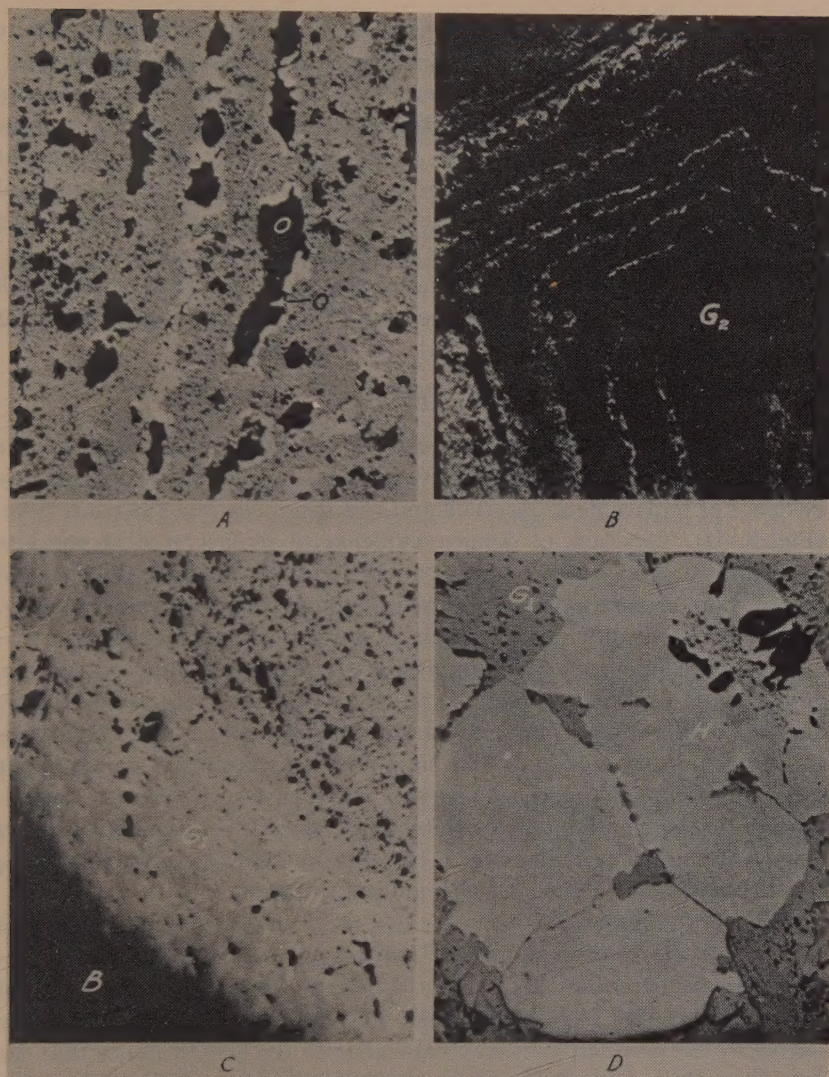


FIG. 3

A. Crystalline hematite (white) partly filling vugs in cryptocrystalline goethite. Plain reflected light. Enlarged 48 diameters.

B. Alternate bands of cryptocrystalline goethite (black) and hematite (white). The goethite is so porous that it has little reflectivity and appears black in the photograph. Plain reflected light. Enlarged 45 diameters.

C. Colloform shell of crystalline goethite lining balsam-filled vug in cryptocrystalline goethite. Same area as in Fig. 2, A. Plain reflected light. Enlarged 54 diameters.

D. Crystalline goethite is apparently replacing the irregular area of hematite along fractures. This is the only suggestion of replacement encountered in the polished sections of the east Texas ores. Plain reflected light. Enlarged 51 diameters.

the material is cryptocrystalline and has a strong purplish-brown to light brown internal reflection which completely masks anisotropism.

Oil immersion of crushed fragments shows that the goethite in the two inner zones is very pure, whereas that of the outer zone contains a large amount of impurity. Material from zone 1 contains less than one per cent of quartz; that from zone 2 not over one per cent of quartz and a trace of the glauconitic mineral; whereas zone 3 contains about 50 per cent iron stained quartz and a small amount of brown clay mineral.

Hematite is present in nearly every section examined. It occurs in all three zones, either as a filling in the vugs (Fig. 3, A) or as narrow bands alternating with goethite (Fig. 3, B).

PARAGENESIS

The paragenetic relationships between goethite and hematite in the brown iron ores of east Texas are not clear. In only one section is any evidence of replacement observed and this is not regarded as conclusive. (Fig. 3, D). This lack of definite evidence of replacement of goethite by hematite or vice versa seems to call for some process which would deposit both minerals contemporaneously or almost contemporaneously. Such a process is difficult to explain on physico-chemical grounds.

The lobate boundary that separates goethite areas of different texture, and goethite from hematite (Fig. 2, C) is a striking feature of the ores. Similar colloform structures have been interpreted¹⁰ as indicating original deposition as a colloid. The flamboyant structure of some of the colloform shells (Fig. 2, A) is believed to indicate subsequent recrystallization.¹¹ The banding of goethite and hematite in the outer zones (Fig. 3, B) appears analogous to banding in manganese oxides where it is believed to result from rhythmic precipitation.¹²

Dehydration may cause hematite to form from goethite and hydration may form goethite from hematite. If either process had been effective to an important extent in these ores it is believed that definite evidence of replacement would have been found. The evidence of contemporaneity offered by the polished sections combined with the field evidence that the ores have been formed by ordinary weathering processes under near surface conditions leads to the conclusion that both the hematite and goethite were deposited at ordinary temperature and that the determining factor was probably chemical.

¹⁰ Cooke, S. R. B., Microscopic structure and concentrability of the important iron ores of the United States: *U. S. Bureau of Mines, Bull.* 391, pp. 52-53, 1936.

¹¹ Cooke, S. R. B., *op. cit.*, pp. 53-56.

¹² Thiel, G. A., The textural relationships of the opaque manganese minerals in Fairbanks, E.E. and others: *Laboratory Investigation of Ores*, pp. 169-171, New York, 1928.

SUMMARY

The foregoing discussion shows that the relations of the iron oxide minerals of the brown ores may be effectively studied in polished sections. The first step in the study of the ores from a district should be the positive identification of the minerals present by means of x -ray diffraction patterns, or the measurement of refractive indices. Once it is known what minerals are present it is usually possible to recognize them in polished sections by ordinary methods.

Significant features of the east Texas ores are as follows:

1. The iron oxide minerals consist of about 80 per cent goethite and 20 per cent hematite. Lepidocrocite is absent. Quartz is the predominant impurity, but the total amount is not large.
2. The presence of both crystalline and cryptocrystalline goethite and hematite.
3. Roughly ellipsoidal structure characteristic of the iron carbonate concretions from which the brown ores have been derived.
4. Prominent colloform structure of both goethite and hematite.
5. Flamboyant internal structure of many of the colloform shells.
6. Banding of goethite and hematite, particularly in the outer zones.
7. Absence of definite evidence of replacement.

It appears that both hematite and goethite are essentially contemporaneous; that both were deposited at ordinary temperatures and that there has been little if any alteration of goethite into hematite, or of hematite into goethite.

This paper is presented with the hope of stimulating further interest in the investigation of the brown iron ores in order that their paragenesis may be definitely established.

MICROCHEMISTRY OF THE PRECIOUS METAL ELEMENTS

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INTRODUCTION

The precious metal elements to be discussed in this paper are platinum, palladium, rhodium, ruthenium, iridium, osmium and gold. Considerable attention has been devoted successfully in the past to means of identifying gold and silver in minerals, but the microchemistry of the remaining six elements has been generally neglected. In part, this neglect results from the complex chemical behavior of these elements and, in part, from the assumption that the platinum-palladium group of minerals are rare. Recent discoveries of noteworthy amounts of several of these elements in many ores emphasize the need of some method whereby the composition of unknown minerals encountered during mineragraphic investigations can be tested for the presence of the precious metal elements.

The need for distinctive microchemical tests is increased because all of these seven elements are known to form natural alloys with one or more of the others, and isomorphism among the proved minerals is known for some of these elements and suspected for others.

The elements mentioned above can be divided into two groups on the basis of their atomic weights and chemical behavior. The silver group—consisting of silver, palladium, rhodium and ruthenium—have atomic weights ranging from 107.8 to 101.7. The gold group contains the heavier elements (gold, platinum, iridium and osmium) with atomic weights between 195.7 and 191.0. Each group shows many chemical similarities but in general it is much more difficult to distinguish between individual members of the two groups than between members of separate groups. Considerable difficulty has been encountered in distinguishing platinum from iridium, or palladium from rhodium. Silver is omitted from the interference studies and discussions because, unlike the other seven elements, it does not form a soluble chloride.

The ideal test would, of course, be one that is distinctive, sensitive and specific for a given element under all conditions and in the presence of any or all of the other elements. Such a test is difficult to obtain, particularly for elements as closely related chemically as those under consideration. In the following discussion, particular attention is given to those tests which are distinctive for one element in the presence of the other elements, and also to the modifying or interfering effect of each of the elements on any test.

Discussion of interferences is confined to the seven elements studied. While it is well known that other elements may occur in the same mineral as several of the elements studied, none of the precious elements, except palladium, is soluble in dilute nitric or dilute hydrochloric acid. Consequently the other base elements, which are soluble in one or other of these acids, can be leached from the decomposed mineral before the precious elements are taken into solution. When palladium is present, a preliminary leaching with *dilute* hydrochloric acid will remove or fix in insoluble form any elements which are likely to be associated with palladium and which might interfere with the dimethylglyoxime test for palladium.

In the course of this study the behavior of many reagents with these elements has been investigated, but only those which give a reliable and distinctive test with one or more of the elements are mentioned in this paper. The reactions between many of these reagents and individual elements have been mentioned or discussed in numerous places in the chemical literature. The author has drawn freely on Chamot and Mason,¹ Behrens and Kley,² Wagner,³ Mellor,⁴ Watson,⁵ and the Abstracts of the American Chemical Society. Many of the previously recommended tests, although entirely satisfactory for a single element, were found to be useless in the presence of any interfering element, either because the test failed to work or because the product was so changed in color, form, or appearance as to be unrecognizable. Consequently, the reagents have been chosen, not only because they give a test with a particular element, but also because that test is distinctive under the microscope and is not seriously affected by the presence of other elements.

MINERALOGY OF THE PRECIOUS METAL ELEMENTS

The following brief summary of the mineralogical occurrences of the precious metal elements is not exhaustive but presents most of the established natural alloys and minerals so that the worker may have a knowledge of the possible combinations of elements and the likely interfering elements encountered in testing for the precious elements.

PLATINUM

Platinum occurs as the native metal, rarely pure, commonly alloyed

¹ Chamot, E. M., and Mason, C. W., *Handbook of chemical microscopy*, New York, 1931.

² Behrens-Kley, *Mikrochemische Analyse*, Voss, Leipzig, 1921.

³ Wagner, P. A., *Platinum deposits and mines of South Africa*, London, 1929.

⁴ Mellor, J. W., *A comprehensive treatise of inorganic and theoretical chemistry*, vol. XV, Longmans, Green and Co., New York, 1936.

⁵ Watson, J. A., Colour reactions in the micro-chemical determination of minerals: *Mineral. Mag.*, vol. 24, No. 148, pp. 21-34, March 1935.

with iron, copper, gold, or other members of the precious metal group. The following tabulation gives many of the native platinum alloys.

TABLE 1*

	Pt	Pd	Rh	Ru	Ir	Os	Au	Fe	Cu
Platinum	100								
<i>a</i> -Ferroplatinum	71-78	0.2-0.8			1-4.5			16-21	
<i>b</i> -Ferroplatinum	73-78							16-20	
<i>a</i> -Polyxene	80-90	0-2.5			1-5.5			6-11	
<i>b</i> -Polyxene	80-90							6-10	
Cuproplatinum	70	0-0.25			1-2			12-15	8-13
Platiniridium	56				4			28	
Iridium	20				77				
Palladic platinum	73-84	3-22			0-3.6				
Rhodic platinum			4.6						
Platinic iridosmium	10.1		1.5		55.2	27.3			
Iridic gold	3.8				30.4		62.1		
Platinic iron	8.2							91.8	

* This table and the following discussion is based mainly on data given in Wagner, *op. cit.*, pp. 11-12, and Mellor, *op. cit.*, pp. 498, 545, 592-93, 686, 730.

Sperrylite (PtAs_2) is the most abundant of the platinum minerals, although Cooperite (PtS) and Braggite (Pt, Pd, NiS) are also found in small amounts.

The solubility of the platinum alloys varies greatly depending on the composition of the alloy. Those containing substantial amounts of iron or copper are fairly readily soluble in aqua regia. Those containing much osmium or iridium are not soluble in aqua regia. The arsenide and sulphide platinum minerals are slightly soluble in aqua regia but the rate of decomposition is very slow. Some sperrylite appears to be untouched by aqua regia. Cooperite is insoluble in aqua regia.

PALLADIUM

Palladium occurs as the native metal but like platinum it is commonly alloyed with other elements. In addition to the palladium-bearing alloys mentioned in Table 1, palladic gold containing 8 to 11.6 per cent palladium, 86 to 91 per cent gold, with small amounts of silver, and potarite—an alloy of palladium and mercury, are also found as minerals.

Stibiopalladinite, Pd_3Sb , is the only known palladium antimonide. Braggite (Pt, Pd, NiS), carries up to 20 per cent palladium. Allopalladium has been reported to contain palladium, silver, gold and selenium. Also, clausthalite is reported to contain mercury and palladium, plumbojarosite up to 0.2 per cent, and covellite may carry some palladium.

Whether the palladium in these last three minerals is present as inclusions or as solid solution is not certain.

Palladium differs from the rest of the precious element group in that several of the palladium minerals are readily decomposed by nitric acid. Palladium, particularly if alloyed with gold or silver, dissolves in dilute nitric acid. Fine grained palladium is soluble in concentrated nitric acid, concentrated hydrochloric acid or aqua regia.

RHODIUM

Little is known concerning the mineralogy of rhodium. In addition to the alloys mentioned in Table 1, rhodite contains 34 to 43 per cent rhodium together with gold and some silver, and rhodium-bearing osmiridium and iridosmium contain up to 17.2 per cent. Most platinum minerals carry some rhodium, in rare specimens the rhodium content reaches 5 per cent. Sperrylite may contain up to 0.72 per cent rhodium.

Metallic rhodium is not soluble in aqua regia unless alloyed with gold, copper, lead, bismuth or platinum.

RUTHENIUM

Ruthenium is present in osmiridium up to 8.5 per cent, in ruthenic osmiridium up to 18.3 per cent, and in ruthenic iridosmium up to 13.4 per cent. The ruthenium sulphide, laurite (RuS_2) or $(\text{RuOs})\text{S}_2$ is also known.

Ruthenium is reported to be soluble in concentrated HCl but laurite is only slightly attacked by aqua regia.

IRIDIUM

The commonest occurrence of iridium is alloyed with osmium to form the natural alloys osmiridium, containing up to 17 per cent iridium, and iridosmium with 47 to 77 per cent iridium. Osmite contains 10 per cent iridium and many of the platinum minerals also carry iridium (Table 1). Iridic gold carries up to 30.4 per cent iridium. No sulphides or arsenides of iridium are known. Most iridium compounds are insoluble in aqua regia.

OSMIUM

All of the important osmium alloys have been already mentioned. No sulphide or arsenide of osmium is known, although laurite may contain up to 3 per cent osmium. Osmium alloys in general are not soluble in aqua regia.

GOLD

Gold occurs in natural alloys with some of the precious metal group to form minerals such as palladic gold (sometimes called porpexite) containing 86 to 91 per cent gold, rhodite (rhodic gold), iridic gold and platinic gold. The ordinary gold minerals are well known. Although most of the gold alloys and minerals are decomposed by nitric acid, the gold is soluble only in aqua regia.

SOLUTION OF SAMPLE

From the preceding discussion it is evident that the precious metal elements and minerals differ greatly in their solubility. Some are soluble in nitric acid, some in aqua regia, and some are practically insoluble in any ordinary acid. The presence of osmium or iridium in a mineral or alloy usually increases its insolubility. All of these eight elements, except silver however, are soluble as chlorides and can be converted into that form by means of the well known method of fusion with sodium chloride in a chlorine atmosphere. This method is best suited to larger quantities of material than are used in ordinary microchemical tests, but by skillful manipulation it is possible to apply the method to very small samples.

For the present investigation, a pure sample of gold chloride was dissolved in distilled water to make a solution containing 1 per cent of the element by weight. Samples of chemically pure platinum, palladium, rhodium, ruthenium, iridium and osmium were treated as follows.

A weighted quantity of the element was ground in an agate mortar with about four times its weight of fused sodium chloride and the mixture placed in a small, hard, open, glass tube. The tube was then connected with a chlorine generator and carefully heated until the mass gently glowed. This treatment was continued for three minutes, then the tube was disconnected and its contents allowed to cool. The contents of the tube were dissolved in distilled water and filtered. Any undissolved residue was again treated in a similar manner. Ordinarily two fusions are sufficient. The resulting solution was then made up to such volume as to contain 1 per cent of the metal by weight. Any silver present is, of course, converted into the chloride, insoluble in acids or water but soluble in ammonium hydroxide.

Avoidance of overheating is necessary, particularly with platinum, the chloride of which is readily decomposed by excess heat. With care, however, all of the elements can be obtained as soluble chlorides by this method which has also been recommended for use in quantitative analysis.⁶ The colors of the solutions may be of some assistance in de-

⁶ U. S. Bureau of Standards Notes. *Jour. Franklin Institute*, vol. 217, Jan. 1934, pp. 106-107.

termining the dominant elements present. Thus the 1 per cent solution of gold is yellow; platinum and osmium, yellow with a faint tinge of orange; iridium, brown; palladium, brownish red; rhodium, cherry red; and of ruthenium, dark cherry red.

PROCEDURE

The elements studied are thus present as compound chlorides of the element and sodium, except gold, which is in solution as gold chloride. The solution of each element in distilled water contains either 1 per cent or 0.1 per cent of the element by weight.

The reagents are used in the strengths indicated in the following section. Potassium iodide and ammonium chloride are used in the solid form. When using potassium iodide, only a small fragment is added to the test drop; with ammonium chloride sufficient quantities are added to give a slight excess of the reagent.

The reactions between individual elements and the reagents are mostly run on a 1 per cent solution of the element. If the element gives a useful reaction, the test is repeated on a 0.1 solution. In the study of interferences, tests are first made using a drop of a 0.1 per cent solution of the element to be tested added to a drop of a 1 per cent solution of the other, possibly interfering, element. If no interference is obtained the test is considered satisfactory. If there is interference, the test is repeated using a 1 per cent solution of each element. If no test is then obtained, the test is considered useless. If, however, this test is free from interference, it is recorded but appears in a different column in Table 2.

The rate of formation of the precipitate is noted in the description of each test. In part this is determined by the concentration of the solution but since all or nearly all of these elements react with many of the reagents, the relative solubility of the precipitates frequently affords a useful distinction. Consequently the relative rapidity of precipitation is a valuable criterion. Accordingly all tests are observed and successive reactions noted until the drop evaporates. While only those tests which give rapid precipitation are recommended, it frequently happens that much additional information concerning other elements in solution can be obtained by observing the test drop until it is dry.

For each reagent studied, the reaction of the individual elements is first presented and is followed by interference reactions for those elements which give a useful precipitate with the reagent.

The following abbreviations and symbols are used throughout the description of tests.

- D. L. Transmitted light. When the color of a precipitate is given without designation, it is to be assumed that the observation was made in transmitted light.

R. L. Reflected light.

(1) A solution containing 1 per cent by weight of the element.

(0.1) A solution containing one tenth per cent by weight of the element.

REAGENTS AND THEIR STRENGTHS

Benzidine	0.05 gm. benzidine dissolved in 10 cc. glacial acetic acid, dilute to 100 cc. with distilled water.
Thiourea	10 per cent solution in water.
Dimethylglyoxime	1 volume of 2 per cent solution in alcohol added to 1 volume of water.
Dimethyl-amino-benzal-rhodanine	0.03 per cent solution in acetone.
Pyridine-hydrobromic acid	1 volume of pyridine added to 9 volumes of 40 per cent HBr. Solution deteriorates rather quickly.
Potassium mercuric thiocyanate	5 per cent solution in water.
Ammonium bichromate	A solution in water of such strength as to give a good silver test with a one tenth per cent solution of silver, i.e., approximately 0.1 gm. of the salt in 30 cc. of water.
Potassium bichromate	A solution of the same color intensity as that for ammonium bichromate.
Potassium iodide	Solid
Ammonium chloride	Solid
Cesium chloride	Solid

DETAILED INVESTIGATION OF TESTS

AMMONIUM BICHROMATE

Individual Reactions

1 per cent solution	
Platinum	Immediate precipitation of feathery crosses, cubes and octahedrons. Dark in D.L., yellow in R.L.
Palladium	Bichromate slowly produces a strong precipitation of feathery crosses and jagged three pointed stars, brown to black in D.L., reddish brown in R.L. Slow but strong test.
Rhodium	No precipitate until drop evaporates.
Ruthenium	Bichromate produces a fairly rapid precipitation of very small red grains, red in R.L.
Iridium	Immediate precipitation of black cubes, cube-octahedron forms, rhombs and crosses, black in R.L. Forms are larger and better developed than with potassium bichromate.
Osmium	Immediate precipitation of orange-to-black rhombs and hexagons.
Gold	No precipitate.

Interference Tests

Pt (1) Ir (1)	Immediate precipitate of orange to black crystals, wine-red in R.L. First crystals precipitated are iridium, then mixed crystals, and finally yellow platinum forms. Strong test.
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AMMONIUM CHLORIDE

Individual Reactions

1 per cent solution	
Platinum	Immediate copious precipitate of feathery crosses, cubes, feathery three, four, and five pointed stars; dark in D.L., very faint yellow in R.L.

Palladium	Slow formation of long yellow needles which in some tests grow across drop. Needles are pleochroic, from golden yellow to grass green; straight extinction, high birefringence.
Rhodium	No precipitate until drop is partly evaporated. Then tiny dark cubes with a high index first form, followed by large pink hexagons and petals. These are usually thin and have a moderate index. Test is very characteristic but slow in forming.
Ruthenium	No precipitate until drop is partly evaporated. Then slowly tiny yellow cubes form and become dark as they grow. Excess reagent becomes stained dark yellow. Later, a few crosses may form and sometimes, just as drop dries, pinkish brown hexagons may appear. Test is not distinctive or reliable.
Iridium	Immediate precipitate of tiny black cubes around reagent. Cubes very small but very abundant. Black in R.L.
Osmium	Immediate and copious precipitation of medium-sized, reddish brown to black cubes, three and four pointed stars, and octahedrons. Reddish brown in R.L.
Gold	No precipitate even in dry drop. Ammonium chloride blades are colored yellow in dry drop.

Interference Tests

Pd (0.1)	Yellow palladium spines form just before drop dries, weak test.
Pd (0.1) Rh (1)	Pink rhodium hexagons slowly form on edge of drop, strong rhodium test, no trace of palladium.
Pd (1) Rh (0.1)	Fairly quick development of good yellow palladium spines. No rhodium test.
Os (0.1) Ru (1)	Add NH_4Cl in excess, rapid precipitation of reddish brown to black cubes and octahedrons; reddish brown in R.L., copious precipitate of osmium. No interference from ruthenium.

BENZIDINE

Individual Reactions

1 per cent solution	
Platinum	Quick formation of long, thin, hairlike needles, green to yellow green, straight extinction, high birefringence in thicker needles; yellowish white in R.L. Needles usually single units, but sometimes several will radiate from a common center. Very distinctive form and test.
Palladium	No immediate precipitate. As drop evaporates, radiating needles and branching blades grow from the edge, showing brownish yellow in D.L., yellow in R.L. Test is slow to start but strong before drop dries.
Rhodium	No immediate precipitate. Slow formation of spherules and rounded grains; pink to yellowish black in D.L., rose pink in R.L. Slow but strong test.
Ruthenium	No immediate precipitation. As drop dries, rounded grains and spherules, or a few needles (sometimes both), all faintly yellowish in D.L., yellowish orange in R.L. form. Slow test, but strong. Forms not very distinctive.
Iridium	Immediate precipitation of small, faint yellow to black, isometric grains with a high index of refraction; light yellow in R.L. Strong test.
Osmium	After about 15 seconds, long, brilliant yellow blades form. Straight extinction, faintly pleochroic, from light to dark yellow. Yellow in R.L.
Gold	Slow formation of yellow needles and clusters around edge of drop. Strong test as drop dries.

Interference Tests

Platinum

- Pt (0.1) Pd (1) Long yellow needles and blades form slowly from edge of drop, later developing into branching blades and typical palladium forms. Platinum test is distinct.
- Pt (1) Pd (1) Immediate precipitation of hair-like needles, followed much later by palladium blades. Strong platinum test.
- Pt (0.1) Rh (1) Yellow needles first grow from edge of drop (typical platinum form), somewhat later there is precipitation of faint pink, rounded grains, high index of refraction. Faint pink also in R.L. Both elements show up clearly.
- Pt (0.1) Ru (1) Yellow platinum needles quickly form from edge of drop. As drop evaporates, rounded grains, faint yellow in D.L., brownish-pink in R.L. slowly form. Ruthenium forms appear in abundance just before drop dries. Satisfactory test for both elements.
- Pt (0.1) Ir (1) Immediate precipitation of small, round-to-irregular shaped grains; very high index; dark in D.L., faint yellow in R.L. Later a few yellowish green blades may form. Strong test, but platinum needles and blades may not appear.
- Pt (1) Ir (1) Same precipitate as above. Platinum needles may or may not appear.
- Pt (0.1) Os (1) Immediate precipitation of yellowish green needles which appear more abundantly and more quickly than from platinum solution alone. No trace of osmium blades.
- Pt (0.1) Au (1) Yellow-to-purple clusters of very small needles and small grains slowly form. Later typical platinum needles grow from edge of drop.

Osmium

- Os (0.1) Rh (1) Quickly forms good rhodium spherules, dark to yellow in D.L., faint pink in R.L. No trace of osmium forms.
- Os (1) Rh (1) Rhodium spherules quickly form, then large yellow osmium blades. Good test for both elements.
- Os (0.1) Pd (1) Slowly, yellowish brown acicular clusters of roughly round shape appear. These are the palladium precipitate, no trace of osmium.
- Os (1) Pd (1) Strong rapid growth of yellow osmium blades, as drop evaporates yellow-brown palladium clusters slowly appear.

Rhodium

- Rh (0.1) Pd (1) A few rhodium spherules are formed, but not until after most of the palladium has precipitated. Test not satisfactory for rhodium.
- Rh (1) Pd (1) Immediate precipitation of rhodium spherules. Strong test. After drop is partly evaporated, greenish-yellow palladium clusters form and partly mask rhodium spherules. Definite test for both elements.
- Rh (1) Pd (0.1) Immediate precipitation of abundant rhodium spherules. Practically no trace of palladium even as drop dries. Since a tenth per cent solution of either rhodium or palladium alone gives a good test, it appears that the element predominating in a test drop determines the form of the precipitate.

CESIUM CHLORIDE

Individual Reactions

1 per cent
solution

- Platinum Immediate precipitation of long, greenish brown needles radiating out from

	grain of CsCl. Needles have many branching arms at right angles. Later feathery crosses, poorly shaped cubes and irregular grains form. All are golden brown in R.L. Isotropic.
Palladium	Immediate fine-grained precipitate, black in D I., yellowish brown in R.L. Isotropic.
Rhodium	Immediate fine-grained precipitate, yellowish brown in D.L., pinkish in R.L. Beyond the ring of fine-grained precipitate, good needles and acicular clumps form. Some of needles are birefringent with parallel extinction.
Ruthenium	Immediate fine-grained precipitate, brownish golden in D.L., same color in R.L.
Iridium	Immediate fine-grained precipitate, brown to black in D.L., pinkish brown in R.L. Later, good hexagons and stars slowly form.
Osmium	Immediate fine-grained precipitate, lemon yellow in center of drop, dark to black on outer edge of precipitate. Feathery crosses and branching forms slowly develop. All lemon yellow in R.L.
Gold	Immediate precipitate of a fine-grained aggregate, greenish yellow to reddish in R.L. Followed by growth of cubes, three and four pointed stars, and octahedrons, dark to yellow brown in D.L., whitish to yellow in R.L. Then slow growth of cube-octahedron crystals, yellow in D.L., dark in R.L., and straw yellow petals and platy forms. All have a high birefringence.

DIMETHYL-AMINO-BENZAL-RHODANINE

Individual Reactions

1 per cent solution	
Platinum	No precipitate.
Palladium	Strong pink color in solution. Immediate precipitation of pink needles and clusters. Presence of 1:5 HCl prevents coloration of solution but not formation of precipitate. Color shows up best in a (0.1) solution.
Rhodium	Color of solution gradually changes from normal faint pink to yellow, with precipitation of tiny pink needles. Reaction best seen in a (0.1) solution.
Ruthenium	Immediate precipitation of colorless to yellowish brown needles, and radiating clusters. Low birefringence. Yellowish in R.L.
Iridium	No definite precipitate.
Osmium	No definite precipitate.
Gold	Immediate precipitation of a purplish fine-grained aggregate, which slowly changes in color to yellow green; then a mass of small yellow green needles form, white in R.L. Still later red hairy clusters, red in R.L. may also appear. Strong test.

Interference Tests

Ruthenium	
Ru (0.1) Pt (1)	Rather slow precipitation of ruthenium needles, some clusters, but most as single units, colorless, faint yellow or faint blue green. Straight extinction, low birefringence. Good test but it forms slowly.
Ru (0.1) Pd (1)	Copious precipitate of fine-grained needles and strong pink color in solution.
Ru (0.1) Rh (1)	Immediate development of a strong pink color in solution followed by precipitation of fine-grained needles, colored pink. Color of solution gradually fades.

Ru (0.1) Ir (1)	Immediate precipitation of needles and clusters of ruthenium. No sign of iridium.
Ru (0.1) Os (1)	Immediate precipitation of ruthenium needles and clusters. No trace of osmium.
Ru (0.1) Au (1)	Immediate precipitation of purple fine-grained aggregate, color gradually turns green and green needles appear. No trace of ruthenium test.
Ru (1) Au (1)	Immediate precipitation of purple to green precipitate, also ruthenium clusters and needles quickly appear. Good test for both elements.

DIMETHYLGLYOXIME

Individual Reactions

1 per cent solution	
Platinum	No precipitate. Drop dries to colorless blades and prisms with blue and yellow interference colors.
Palladium	Immediate heavy precipitation of felted needles, yellow in D.L., pale green with a tinge of yellow in R.L. After standing, the precipitate recrystallizes into larger needles which are darker and greener in D.L., almost colorless in R.L. Dried drop is pinkish.
Rhodium	Precipitation in a few seconds of a very fine-grained mass of small needles, sometimes felted, dark in D.L., greenish yellow in R.L. As drop dries, faintly pink blades and feathery crosses form; needles become greenish. Test very similar to that for palladium except that needles lack yellow color in D.L. Dry drop is pink.
Ruthenium	No precipitate. As drop dries, dark feathery blades form, with black cubes also deposited just before drop is dry. Dry drop is pink.
Iridium	No precipitate. As drop dries, dark blades and stumpy forms are precipitated. Dry drop is colorless.
Osmium	Slowly precipitates yellowish blades and stumpy forms. Dry drop is yellowish.
Gold	Slow precipitation of an aggregate of small needles and stumpy prisms, colorless to faint greenish in D.L., practically colorless in R.L. Needles rarely branch but in general, precipitate cannot be readily distinguished from that of palladium or rhodium if they are present.

Interference Tests

Pd (0.1) Pt (1)	Immediate precipitate of palladium forms. Add benzidine and get good platinum test.
Pd (0.1) Rh (1)	Immediate precipitate but cannot tell whether it is palladium or rhodium. Much of precipitate dissolves in conc. HCl.
Pd (0.1) Ru (1)	Immediate precipitate of palladium forms. Add rhodamine and just before drop dries get a fair Ru test. Drop dries brown.
Pd (0.1) Ir (1)	Immediate precipitate of palladium forms. Add ammonium bichromate. Typical iridium forms appear, but palladium forms slowly disappear and palladium reprecipitates as the chromate.
Pd (0.1) Os (1)	Immediate precipitate of palladium forms. No trace of osmium.
Pd (0.1) Au (1)	Immediate precipitation but cannot tell whether of palladium or gold. Much of precipitate dissolves in conc. HCl.
Pd (0.1) Conc. HCl	Immediate precipitate of palladium forms.

Rh (1) Conc. HCl	No precipitation. Rapid precipitation, however, in presence of 1:5 HCl.
Au (1) Conc. HCl	No precipitation. Rapid precipitation, however, in presence of 1:5 HCl. Palladium can be distinguished from rhodium and gold with dimethylglyoxime by strongly acidifying the test drop with conc. HCl. The addition of 1:5 HCl will not prevent the precipitation of rhodium or gold glyoximes. The rhodium precipitate is readily soluble in conc. HCl; the gold precipitate is slowly soluble and requires a considerable excess of conc. HCl in the test drop.
Pd (0.1) Rh (1) conc. HCl	Immediate precipitation of palladium in about the same quantity as from a drop of (0.1) Pd without acid.
Rh (1) Pd (1)	Add conc. HCl and dimethylglyoxime, good palladium test no rhodium test, filter, and try A or B; A—Add benzidine; slowly get strong test of poorly formed dark spherules and irregular grains, faint pinkish tinge in reflected light. Very strong test but slow. B—Add KI; solution turns reddish brown, immediate precipitation of dark fine-grained aggregate that spreads as a ring from reagent, partly soluble in excess KI. Yellow in reflected light. Forms not distinctive but strong test. C—Add $K_2Hg(CNS)_4$, no precipitate until dry.
Ru (1) Pd (1)	Add conc. HCl and dimethylglyoxime. Good palladium test, filter, A—Add thiourea and heat; strong greenish blue color. B—Add rhodamine; copious precipitate of yellow needles and cubes.
Os (1) Pd (1)	Add conc. HCl and dimethylglyoxime, good palladium test, filter, A—Add thiourea; strong pink color for osmium.

POTASSIUM BICHROMATE

*Individual Reactions*1 per cent
solution

Platinum	Immediate precipitation of yellow cubes, rhombs, and other forms, light yellow color in R.L.
Palladium	Rapid growth of long, colorless to light yellow needles, high birefringence, followed by brown feathery crosses and grains, red in R.L.
Rhodium	Rapid formation of blades (as in palladium test) which seem to break down to give a fine-grained precipitate. As drop evaporates, pink ragged blades form at edge of drop. Sometimes no precipitate is formed.
Ruthenium	Immediate development of a yellowish brown color in test drop. No precipitate until drop is nearly dry, then blades and needles form around edge of drop. No distinctive test.
Iridium	Immediate precipitation of black cubes, rhombs, three pronged stars and cube-octahedron forms. Color black in both D.L. and R.L.
Osmium	With excess of bichromate, precipitation of forms is similar to iridium but red in both D.L. and R.L. Good test but requires either excess of reagent or time for concentration by evaporation.

Gold As drop dries a few spines, slightly darker yellow than bichromate grow in drying drop.

Interference Tests

Ammonium bichromate or

Potassium bichromate 1 per cent solutions of either palladium or rhodium give precipitates with the solid reagent that cannot be distinguished by form or color.

POTASSIUM IODIDE

Individual Reactions

1 per cent
solution

Platinum	Immediate change in color of solution to rich brown followed by rapid precipitation of small black grains which grow to hexagons and rhombs with accompanying bleaching of solution to pinkish or colorless. All crystals are black in R.L.
Palladium	Instantaneous precipitation of dense, very fine-grained, chocolate brown to black powder which spreads as a ring from the KI. Color of powder is black to golden brown in R.L.
Rhodium	Rapid precipitation of needles, radiating clusters, blades and stumpy prisms, solution turns yellow. All forms are highly birefringent, pink in D.L., pink in R.L.
Ruthenium	No precipitation until drop dries, then small yellowish needles form at edge, golden yellow in R.L. Drop dries brown.
Iridium	No precipitate. As drop dries, colorless hexagons grow and slowly stain yellow. Drop dries colorless.
Osmium	Rapid precipitation of small, reddish brown to black cubes and octahedra. As drop dries, brownish blades, crosses and triangular grains grow at edge of drop.
Gold	Drop immediately develops a rich brown to golden yellow color followed by immediate precipitation of a dark fine-grained aggregate yellow to brown in R.L. Later, greenish yellow cubes, petals and other forms develop in brown solution. All of precipitate is a mixture of yellow and brown under crossed nicols. Copious precipitate.

Interference Tests

Pt (1) Ir (1)	Add KI. Strong platinum test. Filter, A—Add ammonium bichromate, good iridium test of red hexagons and black cubes. B—Add benzidine, good yellow spherules, no platinum test.
Os (1) Ir (1)	Add KI. Strong osmium test. Filter, A—Add benzidine, slowly get good iridium test of yellow octahedrons and spherules. Strong test. B—Add ammonium bichromate, weak test and hard to tell iridium forms from osmium-iodide precipitate. C—Add thiourea, exceedingly faint osmium test, practically negative, showing that practically all of osmium was precipitated by the KI.

POTASSIUM MERCURIC THIOCYANATE

Individual Reactions

1 per cent solution	
Platinum	Rapid precipitation of lemon-yellow octahedra and crystals showing cubic and octahedral faces, also some feathery crosses. Isotropic. Color—greenish yellow in thin crystals to black in thick. All colored canary yellow in reflected light.
Palladium	Immediate color change from yellow to yellowish-brown color at junction of drops, followed by precipitation of chocolate brown, rather acicular crystals. Greenish yellow to orange in R.L. As drop dries, pale greenish yellow radiating branches grow.
Rhodium	Immediate precipitation of tiny black grains, white in R.L. As solution dries, branching spines develop with a pale pink color and strong birefringence.
Ruthenium	Slow precipitation of tiny black grains, white in R.L. Test drop turns yellow. Small dark crosses, reddish brown in R.L., form at edge as drop dries.
Iridium	A few small black grains appear but no characteristic precipitate. As drop dries, small pale yellow rectangular needles and crosses form at edge.
Osmium	First forms a few small black cubes, yellowish in R.L. Then slowly at edge of drop, cubes, feathery crosses, dendrites and blades appear, ranging in color from yellow in thin blades to reddish or dark brown in thick crystals. Reddish brown in R.L. No birefringence.
Gold	Immediate copious precipitate of clusters of branching needles, brownish green to brown in direct light, faint yellowish white to reddish in reflected light. Form of precipitate similar to that for copper. Very strong test.

Interference Tests

Pt (1) Ir (1)	Add $K_2Hg(CNS)_4$ —Good platinum test, filter and then add A—Benzidine; strong iridium test of yellow spherules, no Pt test. B—Ammonium bichromate; fair iridium test, no platinum test. Potassium mercuric thiocyanate plus ammonium bichromate—no precipitate.
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PYRIDINE

Individual Reactions

1 per cent solution	
Platinum	No immediate precipitate. As drop dries, yellow prisms, rhombs, and cubes gradually form. Color at first is bright yellow but this gradually deepens to reddish yellow. Pleochroism faint yellow to yellow, moderate index, straight extinction, yellow to reddish yellow in reflected light. Slow but strong test that forms before drop is dry. Insoluble in excess reagent.
Palladium	No immediate precipitate. Very slowly, as drop evaporates, from edge of drop brown rhombs and petals grow into drop. Pleochroic from colorless to brown, straight extinction, abnormal blue interference colors. Strong test but very slow, which forms before drop is dry. Red in reflected light. Forms insoluble in excess reagent.
Rhodium	No immediate precipitate. A few faintly pinkish irregular grains and prisms form just before drop dries. Test practically negative. Forms soluble in excess reagent.

Ruthenium	No immediate precipitate. Slowly as drop evaporates, green cubes, petals and prisms, all very thin, and brown grains and prisms form in drop. Green crystals show deep blue interference colors, blue in reflected light. Red grains are red in reflected light. Strong test but slow; develops in drop before it dries. Insoluble in excess reagent.
Iridium	No immediate precipitate. In drying drop, a few yellow crosses and faintly green cubes form just before drop dries. These are soluble in excess reagent. Test practically negative.
Osmium	No immediate precipitate. As drop evaporates, yellow cubes and petals form around margin. Moderately strong, distinct test. Practically insoluble in excess reagent.
Gold	Immediate copious precipitate of clusters, prisms, needles and feathery forms. Pleochroic from pale yellow to blood red, low extinction, red in reflected light. Strong, distinctive test.

Interference Reactions

Au (0.1) Pt (1)	Immediate precipitation of gold forms. Later yellow platinum prisms and blades envelope the gold crystals. Not soluble in excess reagent. Good test for both elements.
Au (0.1) Pd (1)	Immediate precipitation of gold forms. Considerably later, brown palladium rhombs and petals envelop gold forms. Good test for both elements. Neither precipitate soluble in excess reagent.
Au (0.1) Rh (1)	Immediate precipitation of gold but forms are small and mostly needles. Practically no trace of rhodium until drop dries. Rhodium forms soluble in excess reagent.
Au (0.1) Ru (1)	Immediate precipitation of gold forms. Much later good green ruthenium rhombs and petals envelop gold crystals. Strong test for both elements; neither precipitate is soluble in excess reagent.
Au (0.1) Ir (1)	Immediate precipitation of gold forms. Just before drop dries, a few yellow and green cubes and crosses indicate iridium. These are soluble in excess reagent.
Au (0.1) Os (1)	Immediate precipitation of gold forms. Slowly get yellow osmium cubes, rhombs, prisms and needles. Strong test for both elements; neither precipitate is soluble in excess reagent.

THIOUREA

Individual Reactions

1 per cent solution	
Platinum	Thin yellow petals and rhombs, moderate index of refraction, low birefringence, slowly form. Then jagged prisms grow, dark in D.L., brick red in R.L. Still later long spines grow across drop; dark in D.L., faint yellow in R.L. These usually form just before drop dries.
Palladium	Immediate strong precipitation of fine grained, chocolate brown to black aggregate, followed by very feathery blades and needles that range from light yellow-green to deep yellow in color, high index of refraction, high birefringence. Yellow to pinkish brown in R.L. Strong test. Color of solution changes on addition of thiourea from brown to yellow. Heating the drop dissolves all the precipitate, but it forms again with slow cooling. Rapid cooling gives radiating clusters of (thiourea?) needles stained yellow.
Rhodium	Fairly rapid precipitation of feathery crosses and rosettes of faint to strong yellow color. Very low index of refraction, isotropic. On drying, pink blades

form at edge of drop. Fairly strong test. Gentle heating changes the solution from pinkish to yellowish orange.

Ruthenium	No precipitate. Cold drop very slowly develops a deep inky blue color. As drop dries, blue-black spherules form at edge of drop. If drop is gently heated after thiourea is added, it immediately turns inky blue. Strong, positive test.
Iridium	No precipitate. No color change in cold or hot solution.
Osmium	No precipitate. Cold drop dries to give thiourea blades stained yellow. Gently heated drop after addition of thiourea gives an immediate change to a reddish purple. Very distinctive test.
Gold	Immediate precipitate of blue black grains, soluble in excess thiourea. As drop evaporates, colorless rhombs and petals form at edge, low birefringence. Still later prisms appear with a high birefringence.

Interference Reactions

Ruthenium	
Ru (0.1) Pt (1)	Warmed—Faint but distinct greenish blue color, blue green on drying, green in R.L. No precipitate of platinum forms.
Ru (1) Pt (1)	Warmed—Strong blue color.
Ru (0.1) Pd (1)	Cold—Solution turns yellow and gives yellow feathery blades and needles of palladium test. Warmed—Solution turns greenish blue and forms a few blue spherules on cooling. Test for ruthenium distinct but not striking.
Ru (1) Pd (1)	Cold—Strong test for palladium. Later get blue spherules. Drop dries greenish-blue. Warmed—Solution turns intense blue-green, dries blue-green.
Ru (0.1) Rh (1)	Cold—Light yellow, feathery crosses and clusters of petals slowly form. Good ruthenium test Warmed—Solution turns intense blue green.
Ru (0.1) Ir (1)	Cold—No precipitate, drop slowly loses all color. Warmed—Solution turns intense blue. Dries blue.
Ru (0.1) Os (1)	Cold—No precipitate. Drop remains yellow and slowly develops a few blue spherules. If dry drop is heated it first turns blue and then purple. Strong heating turns it black. Warmed—Drop first turns blue, then purple to red. Dries reddish purple.
Ru (0.1) Au (1)	Cold—Immediate purple gold precipitate which dissolves, leaving solution with a faint blue color. Warm—Strong blue ruthenium color, disappears with overheating.
Osmium	
Os (0.1) Pt (1)	Cold—No precipitate, no color change. Warmed—Drop evaporates to a yellow residue which on stronger heating turns intense pink.
Os (0.1) Pd (1)	Cold—Solution turns greenish yellow, followed by heavy precipitate of palladium forms. Warmed—Palladium forms dissolve, then reprecipitate as drop dries. Dry drop is yellow, strong heating turns it black. No osmium test.
Os (1) Pd (1)	Cold—See above. Warmed—Drop turns pink as it dries, continued heating turns it dark red, then black. Satisfactory osmium test.
Os (0.1) Rh (1)	Cold—Slowly, abundant light yellow feathery crosses appear. Drop retains a faint pink color.

- Warmed—Solution turns orange color, dries orange. Rhodium and thiourea when evaporated to dryness give a residue colored a faint yellow-orange.
- Os (1) Rh (1) Warmed—Solution turns reddish purple, dries purple.
- Os (0.1) Ru (1) Warmed—Solution turns blue, then purple.
- Os (0.1) Ir (1) Cold—No precipitate, no color change.
Warmed—Solution turns pink.
- Os (0.1) Au (1) Cold—Gold precipitate forms and redissolves leaving a colorless solution.
Warm—Strong pink osmium color.

MISCELLANEOUS

- Pd (0.1) Cold—Distinct canary yellow color, precipitation of feathery yellow crosses.
- Rh (1) Very weak precipitate of feathery yellow crosses, solution is pink in cold, changes to yellow on warming.
- Pd (0.1) Rh (1) Cold—Solution immediately turns canary yellow and rapid precipitation of many feathery yellow crosses. Color of solution clearly proves presence of palladium.
- Ir (1) No precipitate with thiourea, add ammonium bichromate—immediate fine grained black precipitate plus green clusters and radiating needles as drop dries.
Thiourea plus ammonium bichromate—No precipitate.

TABLE 2. SUMMARY OF TESTS FOR PRECIOUS METAL ELEMENTS

Element	Test	Distinctive in ten times as much of	Distinctive in equal proportions of	Not distinctive in presence of
Platinum	Benzidine Potassium Iodide	Pd, Rh, Ru, Os, Au Ir, Rh, Ru		Ir Pd, Os, Au
Palladium	Dimethylglyoxime plus conc. HCl Thiourea	Pt, Rh, Ru, Ir, Os, Au Pt, Ir, Rh, Ru, Os, Au		
Rhodium	Dimethylglyoxime Benzidine	Pt, Ir, Os, Ru Ir, Au	Pt, Pd, Os	Pd, Au Ru
Ruthenium	Thiourea Rhodanine	Pt, Rh, Ir, Os, Au Pt, Ir, Os	Pd Au	Pd, Rh
Osmium	Thiourea Benzidine Ammonium bichromate	Pt, Ir, Au Rh, Au	Pd, Rh, Ru Pd, Rh, Ru, Au	Pt, Ir Pt, Pd, Ir, Ru
Iridium	Ammonium chloride Potassium bichromate	Pd, Rh, Ru, Au Pd, Rh, Ru, Au	Pt, Os Pt, Os	
Gold	Pyridine in HBr	Pt, Pd, Rh, Ru, Ir, Os		

RECOMMENDED TESTS

Platinum: The benzidine test gives satisfactory results in the presence of any of the precious elements except iridium. In the presence of iridium, the test is sometimes satisfactory but often all that is obtained is an aggregate of small, shapeless grains with no diagnostic features. Potassium mercuric thiocyanate or potassium iodide will give a distinctive platinum precipitate in the presence of iridium. Iridium can be identified in the presence of platinum, and the remainder of the group, except ruthenium, by first adding KI or potassium mercuric thiocyanate to the drop, filtering and then precipitating the iridium with either benzidine or ammonium bichromate.

Palladium: The dimethylglyoxime test for palladium is both sensitive and diagnostic in the presence of any of the elements considered, providing the test drop is strongly acidified with concentrated HCl. A drop of acid as large as the test drop is sufficient to prevent the precipitation of rhodium or gold glyoxime. In weakly acid or neutral solutions the rhodium and gold glyoximes cannot be distinguished from that of palladium.

Rhodium: Rhodium can be identified in the presence of platinum, iridium, osmium and ruthenium by dimethylglyoxime added to a neutral or weakly acidic solution. Palladium and gold will also be precipitated. A drop of concentrated HCl added to the precipitate, will dissolve the rhodium glyoxime readily, the gold slowly, and the palladium glyoxime not at all. Benzidine will give a distinctive test for rhodium in the presence of gold and iridium. The existence of rhodium in the presence of palladium can be confirmed by first precipitating the palladium with dimethylglyoxime in a solution strongly acidified with HCl solution, filtering and testing for rhodium with benzidine. The precipitate is slow in forming but a good test can be obtained with one tenth per cent of rhodium.

Ruthenium: Thiourea gives a satisfactory test for ruthenium in the presence of any of the group except palladium and possibly osmium. An experienced observer can recognize the ruthenium test in the presence of osmium but the blue ruthenium color is quickly replaced by the red osmium color test. Rhodanine gives a satisfactory ruthenium test in the presence of osmium. If palladium is present, it can be removed with dimethylglyoxime, the solution filtered and ruthenium tested for with either thiourea or rhodanine.

Osmium: Platinum, iridium and gold do not interfere with the thiourea test for osmium, but palladium, rhodium and ruthenium prevent a satisfactory test on one tenth per cent osmium solution when they are present in proportions of one per cent or greater. If the concentration

of osmium is equal to that of these elements, the test is satisfactory. Palladium can be first removed by dimethylglyoxime. Ammonium chloride will give a good osmium test in the presence of ruthenium. Ammonium bichromate or ammonium chloride will prove osmium in the presence of rhodium.

Iridium: Iridium is the most difficult of this group of elements to easily identify, since with most reagents it gives a poorly formed precipitate or no precipitate. Ammonium chloride or ammonium bichromate give a good iridium test in the presence of all these elements except platinum and osmium. If either platinum or osmium are present, they must first be removed by precipitation with potassium iodide, the drop filtered and iridium precipitated by either benzidine or ammonium bichromate.

Gold: The pyridine test for gold is entirely satisfactory in the presence of any of the other precious elements. Moreover, several of the other elements slowly give good tests also with pyridine, but since the gold test forms immediately there is no interference.

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GRAFTONITE FROM GREENWOOD, MAINE*

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ABSTRACT

A specimen of graftonite from Greenwood, Maine, permitted a detailed study of this little known mineral, and established a third locality for this iron, manganese, calcium phosphate.

The optical properties of the Greenwood material showed a slightly higher refringence than the type material from Grafton, N.H., but the birefringence, 0.024, and the optical axial angle, $+2V\ 55^\circ$, are the same. Indices: $\alpha=1.709$, $\beta=1.714$, $\gamma=1.733$.

Chemical analysis shows an isomorphous variation in the proportions of iron, manganese, and calcium, but conforms to the established formula for graftonite, $3(\text{Fe, Mg, Ca})\text{O} \cdot \text{P}_2\text{O}_5$.

INTRODUCTION

During the summer of 1936 Mr. Ernest W. Sniffen of Hampton, Virginia, obtained a specimen of a mineral thought to be triphylite, which he submitted to the U. S. Geological Survey for identification. Optical observations (by J. J. G.) indicated that the mineral was graftonite, and qualitative chemical tests (by J. J. F.) showed it to be an anhydrous iron, manganese, calcium phosphate. Through the courtesy of Mr. Sniffen, permission was granted to make a detailed study of the specimen.

This new occurrence establishes a third locality for graftonite, which has previously been recorded from only two localities, both in Grafton County, New Hampshire. The original locality described by Penfield¹ is the Melvin Mountain mine near the village of Grafton, and the other locality described by Berman² is the Palermo mica mine on Bald Face Mountain, one and a half miles south of North Groton.

The specimen described in this paper came from the Tamminen-Wassinen Ledge, on Noyes Mountain, near Greenwood, Maine. The geology, and a great number of the minerals from that locality were described by Landes³ in his paper on pegmatites of central Maine. The graftonite is massive, showing no crystal faces, and the specimen measures about 2.5 by 2 by 1.5 cm. On one side is a compact assemblage of quartz, albite, muscovite, chlorite, calcite, pyrite, sphalerite, and arsenopyrite, with an occasional grain of triphylite.

* Published by permission of the Director, U. S. Geological Survey.

¹ Penfield, S. L., On graftonite, a new mineral from Grafton, New Hampshire, and its intergrowth with triphylite: *Am. Jour. Sci.*, vol. 9, p. 20, 1900.

² Berman, Harry, Graftonite from a new locality in New Hampshire: *Am. Mineral.*, vol. 12, p. 170, 1927.

³ Landes, K. K., The paragenesis of the granite pegmatites of central Maine: *Am. Mineral.*, vol. 10, pp. 399-411, 1925.

PHYSICAL AND OPTICAL PROPERTIES

The graffonite is homogeneous, ocher-salmon colored, and unaltered except for a few patches of dark staining from oxidation of the iron and manganese. The luster is vitreous to resinous. In appearance it resembles salmon-colored varieties of lithiophilite and triphylite. The graffonite shows no response to ultra-violet radiation.

Unlike the graffonite described by Penfield and Berman, the specimen from Maine does not show on visual inspection the banded structure due to alternating layers of graffonite and triphylite. Microscopic examination of a polished surface by reflected light, however, shows a few sets of very narrow parallel bands of another mineral which in thin section and in crushed fragments are seen to have the optical properties of triphylite. These bands of triphylite are very insignificant and are present only in parts of the graffonite.

The specific gravity of the graffonite was found to be 3.771 on massive material and 3.796 (at 25°C.) on a powdered sample, which is somewhat higher than that (3.672) reported by Penfield. The hardness is between 4 and 5.

Before the blowpipe flame the graffonite fuses easily to a glassy black bead, feebly magnetic. Heated in the closed tube, only a slightly visible quantity of water is given off, and at red heat the mineral fuses. It is readily soluble in cold 1:1 hydrochloric acid, cold 1:1 nitric acid and hot 1:1 sulphuric acid.

Although neither Penfield nor Berman make any mention of cleavage, close inspection of crushed fragments reveals a small proportion of flat cleavage plates, and on a few of these a straight edge indicates a second and less perfect cleavage. A thin section of unknown orientation crystallographically, but almost normal to the negative obtuse bisectrix, shows a number of well developed continuous cleavage lines and a second set of much poorer and interrupted cleavage lines oblique to the other cleavage. The second poorer cleavage is also shown on the right side of Berman's Fig. 1. The optic axial plane is oblique to the traces of both cleavages, as seen in this thin section. The few minute bands of triphylite are nearly parallel to the better cleavage.

The optical properties of the graffonite indicate monoclinic symmetry, as extinction on straight-edged cleavage fragments is parallel on some and inclined 30° on others. The plane of the optic axes is nearly parallel to some elongated cleavage pieces, as seen in crushed material. The positive acute bisectrix, *Z*, is not quite normal to the cleavage. Observed

2V is about 55° to 60°; calculated 2V = 55°. The indices of refraction are: $\alpha = 1.709$, $\beta = 1.714$, $\gamma = 1.733$, B = .024. (See Table 1.)

TABLE 1. COMPILATION OF AVAILABLE OPTICAL DATA ON GRAFTONITE

Locality	Grafton, N.H. Melvin Mt.	Grafton, N.H. Melvin Mt.	Groton, N.H. Baldface Mt.	Greenwood, Me. Noyes Mt.
Data by	Penfield	Larsen	Berman	Glass
Color	Salmon	Salmon-pink	Salmon	Ocher-salmon ^a
α	—	1.700	1.704	1.709
β	—	1.705	1.706	1.714
γ	—	1.724	1.725	1.733
$\gamma - \alpha$	not high	.024	.021	.024
Sign	+	+	+	+
2V	50°-60°	55° ±	50° ±	55°
Disp.	Distinct	Distinct	$r > v$	$r > v$

^a Ridgway Color Standard.

The indices of refraction of the material from Greenwood, Maine, are somewhat higher than those of the two samples from Grafton County, New Hampshire. The birefringence of the Greenwood material (0.024) agrees with that determined by Larsen, but is somewhat higher than the value shown by Berman, who gives $\gamma - \alpha$ as 0.021, and 2V = 50°. This angle gives a calculated value of 0.004 for $\beta - \alpha$, whereas his value is 0.002. This suggests that his α value is a little high. The material from Groton, New Hampshire, is described by Berman as having the same banded structure and association with altered triphylite that characterizes the type material from Grafton, New Hampshire, upon which Larsen made his optical determinations. No analysis was made of the Groton material described by Berman, but the optical properties indicate that it is similar in composition to the original graftonite from the type locality.

The fact that the indices of refraction of the graftonite from Greenwood, Maine, are consistently higher than those for the other two specimens is due to the higher manganese and lower calcium content of the mineral.

CHEMICAL ANALYSIS

The purity and abundance of material from Greenwood, Maine, favored a careful and accurate analysis. Selected, fresh, unstained material was used for the analysis and the results are given in Table 2.

TABLE 2. CHEMICAL ANALYSIS OF GRAFTONITE FROM GREENWOOD, MAINE
(Joseph J. Fahey, analyst)

	Per cent	Molecular ratios
P ₂ O ₅	40.03	.2819 or 1.000
Al ₂ O ₃	none	
Fe ₂ O ₃	none	
TiO ₂	none	
FeO	27.78	.3867
MnO	25.48	.3592
CaO	4.71	.0840
MgO	none	.8454 or 2.999
K ₂ O	0.05	.0005
Na ₂ O	0.16	.0026
Li ₂ O	0.37	.0124
H ₂ O	0.60	
CaCO ₃	0.46	
Insol.	0.18	
F	none	
	99.82	

The ratio RO:P₂O₅ is 2.999:1.000 or 3:1.

The formula of the mineral is $3(\text{Fe, Mn, Ca})\text{O} \cdot \text{P}_2\text{O}_5$.

Ferrous iron was found to be equivalent to the total iron. This indicates that all the iron is in the ferrous state and that there is none other than divalent manganese in the mineral. Manganese was determined volumetrically in a separate portion by the bismuthate method. The blank correction was equivalent to 0.40 cc. of 0.05 N. permanganate solution. The absence of Al₂O₃ was definitely established by two methods, namely the sodium hydroxide method and the hydrochloric acid-ether method. An excess of ferric nitrate was added to the hydrochloric acid solution of another portion in order to hold P₂O₅ in the ammonia precipitate and allow calcium to remain in solution, where after filtration it was determined in the usual manner. The alkalis were determined by the J. Lawrence Smith method, separating LiCl with ether-alcohol solution as recommended in the Palkin⁴ method and modified by Wells and Stevens.⁵ Water was determined by the Penfield method, using sodium tungstate as a flux.

Although Penfield's analysis of graftonite from Melvin Mountain shows a ratio of CaO to (FeO+MnO) close to 1:4 (0.1645:0.6755 or 1.00:4.11), the analysis of the graftonite from Maine, with a lower per-

⁴ Palkin, J., *Am. Chem. Soc.*, vol. 38, p. 2326, 1916.

⁵ Wells, R. C., and Stevens, R. E., *Ind. and Eng. Chem., Anal. Ed.*, vol. 6, p. 439, 1935.

centage of CaO (only about half as much) shows that the quantity of CaO is not constant and that all the bases should be grouped together.

The three analyses of graftonite, from two localities, are shown in the following compilation (Table 3).

TABLE 3. COMPILATION OF CHEMICAL ANALYSES OF GRAFTONITE

	1.	2.	3.
P ₂ O ₅	41.20	40.80	40.03
Al ₂ O ₃	—	—	none
Fe ₂ O ₃	—	10.16	none
TiO ₂	—	—	none
FeO	30.65	24.28	27.78
MnO	17.62	15.38	25.48
CaO	9.23	7.25	4.71
MgO	0.40	—	none
K ₂ O	—	0.14	0.05
Na ₂ O	—	1.15	0.16
Li ₂ O	0.33	—	0.37
H ₂ O	0.75	1.17	0.60
CaCO ₃	—	—	0.46
Insol.	—	—	0.18
F	—	—	none
	100.18	100.33	99.82
G.	3.672	—	3.796 (powder) 3.771 (massive)

1. Graftonite from Melvin Mt., Grafton County, New Hampshire. S. L. Penfield, analyst.
2. Graftonite (partially purified), Melvin Mt., Grafton County, New Hampshire. W. E. Ford, analyst.
3. Graftonite from Greenwood, Oxford County, Maine. J. J. Fahey, analyst.

A RARE-ALKALI BIOTITE FROM KINGS MOUNTAIN, NORTH CAROLINA¹

FRANK L. HESS² AND ROLLIN E. STEVENS³

Several years ago, after Judge Harry E. Way of Custer, South Dakota, had spectroscopically detected the rare-alkali metals in a deep-brown mica from a pegmatite containing pollucite and lithium minerals, in Tin Mountain, 7 miles west of Custer, another brown mica was collected, which had developed notably in mica schist at its contact with a similar mass of pegmatite about one half mile east of Tin Mountain. J. J. Fahey of the United States Geological Survey analyzed the mica, and it proved to contain the rare-alkali metals⁴ and to be considerably different from any mica theretofore described. Although the cesium-bearing minerals before known (pollucite, lepidolite, and beryl) had come from the zone of highest temperature in the pegmatite, the brown mica was from the zone of lowest temperature.

The occurrence naturally suggested that where dark mica was found developed at the border of a pegmatite, especially one carrying lithium minerals, it should be examined for the rare-alkali metals. As had been found by Judge Way, spectroscopic tests on the biotite from Tin Mountain gave strong lithium and rubidium lines, and faint cesium lines. Lithium lines were shown in a biotite from the border of the Morefield pegmatite, a mile south of Winterham, Virginia, but rubidium and cesium were not detected. Similarly placed dark micas from Newry and Hodgeon Hill, near Buckfield, Maine, gave negative results. They should be retested. Tests by Dr. Charles E. White on a shiny dark mica from the Chestnut Flat pegmatite near Spruce Pine, North Carolina, gave strong lithium and weaker cesium lines. Biotite from the Corner Rock pegmatite near Craggy Gardens, North Carolina, was found by Morris Slavin of the Bureau of Mines to give strong lithium lines.

When opportunity was recently had to examine spodumene pegmatites enclosed in mica schists on the W. A. Ware farm, about $2\frac{1}{2}$ miles southwest of Kings Mountain, North Carolina, a lenticular mass of bright brownish mica 4 inches thick, 8 inches long, and 6 inches broad was found. It was on the surface close to the edge of the pegmatite. It is intergrown with pegmatite of border phase (See Fig. 1), and undoubtedly

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² Principal Mineral Technologist, U. S. Bureau of Mines.

³ Assistant Chemist, U. S. Geological Survey.

⁴ Hess, Frank L., and Fahey, J. J., Cesium biotite from Custer County, South Dakota: *Am. Mineral.*, vol. 17, pp. 173-176, 1932.

came from the schist at the contact. None has been found at any place in the pegmatite. Keith and Sterrett⁵ noticed such segregations and said: "The reaction of wall rock and dike is also evident by the unusual coarseness of the mica schist of the wall rock in many places."

There is some white mica in the mass which is easily separated, and in this R. C. Wells, on testing with a pocket spectroscope, found no lithium or rare-alkali metal lines.

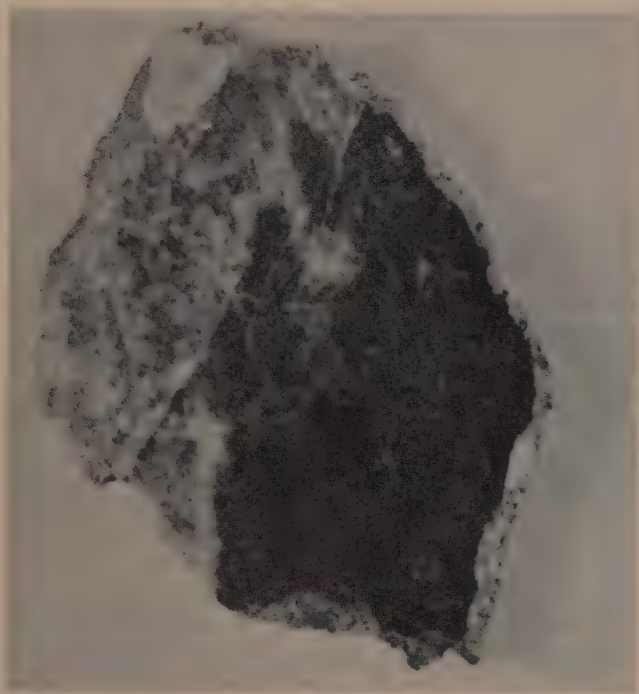


FIG. 1. Intergrowth of rare-alkali biotite with pegmatite, apparently from edge of dike, Ware farm, Kings Mountain, N.C.

In sections under the microscope the brown mica is found to be remarkably pure, except for numerous microscopic prisms of apatite which pierce the mica plates. It does not enclose zircon and shows no halos as does the mica from the vicinity of Tin Mountain; and although both carry considerable titanium, in neither has rutile been found in the plates.

⁵ Keith, Arthur, and Sterrett, Douglas B. Gaffney, Kings Mountain, South Carolina-North Carolina, *U. S. Geol. Survey Atlas* No. 222, 1931, p. 11.

The optical properties, determined by Miss Jewell J. Glass of the U. S. Geological Survey, are as follows:

Optically negative, acute bisectrix X is sensibly normal to the basal cleavage (001). The optical angle (2V) is very small, 0° to 5° . The indices of refraction are: $\alpha=1.555$, $\beta=1.589$, $\gamma=1.590$. Color dark brown. Luster bronzy. Pleochroism along X, pale pinkish brown; Y and Z dark brown to dark reddish brown. Absorption strong, Y and Z > X.

The pegmatite with which the mica is associated is rather remarkable. Dark minerals are exceedingly scarce—even black tourmaline is rare. No massive segregations of mica, feldspar, or quartz; no graphic granite, and almost no beryl were found. The pegmatite was formed in several stages. A comparatively fine-grained mass of quartz and microcline, lean in muscovite, represents the first stage. It was roughly sheeted and long flat bodies of white microcline were introduced, free from perthitic structure, and as much as 2 or 3 feet thick. Sheetting took place again, the feldspar was broken, and later solutions replaced the corners with fine-grained albite, quartz, muscovite, and spodumene. Spodumene seems to make up 15 or 20 per cent of much of the mass. Some cassiterite is present, mostly in greisenized parts of the pegmatite. Graton⁶ reported finding lithiophyllite and lepidolite, but, excepting the mica described here, spodumene is the only mineral carrying a rare-alkali metal that was found in several visits to the deposits. The pegmatites are from 3 to more than 100 feet broad, extend for $4\frac{1}{2}$ miles southwest of Kings Mountain and toward the northeast are known at a number of places between Kings Mountain and Lincolnton.⁷

CHEMICAL ANALYSIS

As the analysis of the mica involved the use of some new methods, a brief outline of the analytical procedure seems desirable.

Silica and fluorine in a gram sample were separated as described by Hoffman and Lundell,⁸ involving fusion with sodium carbonate, leaching of the melt with water, and the separation of the silica by two precipitations with zinc oxide. Two fusions and leachings were made to insure complete removal of fluorine. Silica was determined on the combined residues and zinc precipitates by the usual method.

⁶ Graton, Louis C., Reconnaissance of some gold and tin deposits of the southern Appalachians: *U. S. Geol. Survey, Bull.* **293**, p. 38, 1906.

⁷ For a more detailed description of the pegmatites see: Hess, Frank L., Lithium in North Carolina: *Eng. and Min. Jour.*, vol. **137**, pp. 339–342, July 1936.

⁸ Hoffman, J. I., and Lundell, G. E. F., Determination of fluorine and of silica in glasses and enamels containing fluorine: *Bur. Standards Jour. Research*, vol. **3**, p. 581, 1929.

The fluorine was determined nephelometrically. The extracted fluoride solution, free from silica, was made up to a definite volume with a sodium chloride concentration of 55 grams per liter. In a 25 milliliter aliquot of this solution the fluorine was precipitated as colloidal calcium fluoride, the solubility being reduced by alcohol, and gelatin serving as a stabilizer. A standard of known fluoride content was precipitated at the same time and the two opalescent solutions compared in a nephelometer to give a preliminary figure for fluorine, and from this the unknown and standard were adjusted to within 5 per cent of equality and new aliquots precipitated to obtain a final reading. The nephelometric procedure for fluorine has been shown⁹ to be accurate and dependable.

The rare alkalies were determined by methods described by Wells and Stevens.¹⁰ The alkalies in a 0.5 gram sample were extracted by the J. Lawrence Smith procedure, and potassium, rubidium, and cesium separated as chloroplatinates insoluble in 80 per cent alcohol. From the soluble portion, containing sodium and lithium, platinum was removed with formic acid and sodium and lithium separated by the Palkin method,¹¹ based on the insolubility of sodium chloride in an alcohol-ether solution. The sodium and lithium were then separately weighed as sulphates.

The insoluble chloroplatinates of potassium, rubidium, and cesium were treated with formic acid to remove platinum, the resulting chlorides evaporated to dryness and a number of extractions made with alcohol saturated with hydrogen chloride (10 ml. solvent used) to remove all of the rubidium and cesium. In the first two extracts cesium was removed by precipitating potassium and rubidium with an alcoholic ammonium sulphate reagent. The cesium was then weighted as sulphate and the rubidium in the first two alcohol-hydrochloric acid extracts obtained by difference. From the additional extracts rubidium was obtained by converting to chloroplatinates and extracting with 15 per cent alcohol, the potassium salt being soluble.

This description gives the method of analysis in brief. Details of procedure may be found in the articles cited.

Results of the analysis of the brown mica from Kings Mountain and a corrected analysis of the biotite from the edge of the pegmatite near Tin Mountain, South Dakota, are given below.

⁹ Stevens, Rollin E., Nephelometric determination of fluorine: *Ind. Eng. Chem., Anal. Ed.*, vol. 8, pp. 248-252, 1936.

¹⁰ Wells, Roger C., and Stevens, Rollin E., Determination of the common and rare alkalies in mineral analysis: *Ind. Eng. Chem., Anal. Ed.*, vol. 6, pp. 439-442, 1934.

¹¹ Palkin, S., Separation of lithium from the other alkali metals: *Jour. Am. Chem. Soc.*, vol. 38, p. 2326, 1916.

ANALYSES OF BIOTITE

	1.	2.
SiO ₂	42.02	39.97
Al ₂ O ₃	18.75	17.51
Cr ₂ O ₃	—	none
Fe ₂ O ₃	0.66	2.26
FeO	8.29	14.81
MnO	0.27	0.22
MgO	9.55	8.45
CaO	0.93	none
Li ₂ O	1.20	0.65
Na ₂ O	0.73	0.45
K ₂ O	8.54	8.48
Rb ₂ O	1.85	1.48
Cs ₂ O	0.47	1.12
H ₂ O-110°C.	0.16	0.32
H ₂ O+110°C.	2.44	2.48
TiO ₂	1.35	2.64
F	4.34	3.17
	101.55	101.01
Less O=F ₂	1.83	1.34
	99.72	99.67

1. From Kings Mt., N.C., analysis by R. E. Stevens.

2. From near Tin Mt., S. D., analysis by J. J. Fahey with redeterminations of lithium, rubidium, and cesium by R. C. Wells and R. E. Stevens.

The two micas have a very similar composition. The most marked differences are in the iron and silica. The mica from the light-colored pegmatite of North Carolina, as might perhaps be expected, carries much less iron than that from the more highly colored rocks of the Black Hills, and it makes up the difference in silica. The alkali oxides amount to 12.79 per cent in the Kings Mountain mica, and to 12.18 per cent in the Custer mica, a ratio of 1.05 to 1; the molecular ratio rises to 1.18 to 1, owing to the large quantities of the lighter alkali metals in the southern mica. Each carries less than half the magnesia of an ordinary phlogopite; the iron, even in the South Dakota mica, is low for lepidomelane and both seem to fit best among the biotites. Rare-alkali micas of this kind seem generally to have a high fluorine content, and conversely those low in fluorine do not contain rare alkalies. A black biotite of similar occurrence, collected by W. T. Schaller at Ridgway, Va., contained only 1.40 per cent fluorine and no rare alkalies.

CONTEMPORANEOUS CRYSTALLIZATION OF BERYL AND ALBITE VS. REPLACEMENT

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In many mineral deposits containing the platy variety of albite, one commonly finds beryl and albite in close genetic relationship which, during the past two decades, has been explained by some students of mineral genesis in pegmatites as due to replacement of the beryl by albite, or by the replacement of the albite by beryl.¹ The later mineral, in either case, being introduced in late hydrothermal or pneumatolytic solutions.

During the past five years I have collected specimens of these two intergrown minerals from the Golding-Keene mine, three miles north of Gilsum, New Hampshire, and the Strickland mine on Collins Hill, four miles northeast from Middletown, Connecticut. The beryl-albite intergrowths from these widely separated mineral deposits are excellent examples and are characteristic of this type of occurrence. These intergrowths also possess characters which, without doubt, show the associated minerals to be of essentially contemporaneous origin instead of either one replacing the other.

The beryl crystals containing the intergrown albite are invariably tapered, Fig. 1, and have angles between the opposite prism faces up to 30°. They vary in color from translucent yellow and white to an aquamarine bluish-green at the small end where the material is sometimes

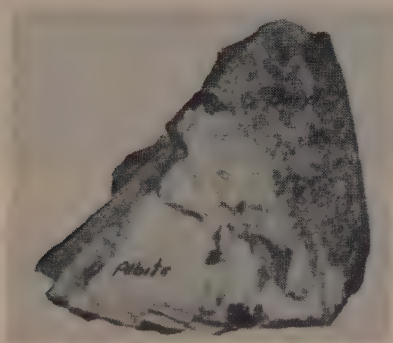


FIG. 1. Tapered crystal of the beryl-albite intergrowth; at the larger end cleavelandite is shown growing *away* from the intergrowth. $X=0.28$. Specimen from the Strickland mine.

¹ Hess, F. L., Pegmatites, *Econ. Geol.*, vol. 28, pp. 447-462, 1923.

Hess, F. L., The pegmatites of the Western States in the Lindgren volume, Ore deposits of the Western States, *A.I.M.E.*, New York, 1934.

Anderson, A. L., Genesis of the mica deposits of Latah County, Idaho: *Econ. Geol.*, vol. 28, pp. 41-58, 1933.

Megathlin, G. R., The pegmatites of the Gilsum area, New Hampshire: *Econ. Geol.*, vol. 24, pp. 163-181, 1929.

clear and transparent but badly fractured. The faces of these crystals, while not rough, lack the high polish frequently found on beryl and show the effects of interference from contemporaneous minerals then crystallizing from the same solution.

At the base or larger end of these tapered crystals where the beryl and albite are most intricately intergrown, the percentage of albite is always the higher. As the growth of the crystal proceeds outward at this end the amount of beryl rapidly diminishes and finally disappears leaving the albite to continue its growth. The large end of these crystals may at times consist of a hexagonal shell of beryl surrounding a "core" of fine-grained albite. Close, parallel sections show that the albite soon occurs as an intergrowth as illustrated in Fig. 2. The beryl at the large ends consists of plate or blade-like masses usually having their sides parallel to the first or second order prisms. In addition other forms as pyramids and pinacoid were noted. The luster of these is of the same quality as that of the external faces of the crystals.

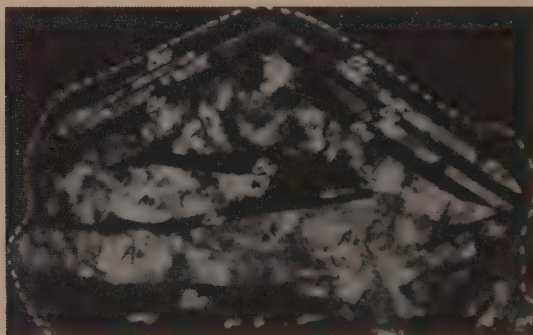


FIG. 2. Section perpendicular to the c axis of a tapered beryl-albite crystal from the Golding-Keene mine. Albite (Ab), Beryl (Be). $X=1.4$. (Dotted line shows the outline of the section.)

The intergrowths break apart very readily and expose many contacts between the beryl and albite which show clearly that the latter was moulded around the beryl.

Within the crystal the albite is usually irregularly arranged and is often close to one side instead of being centrally located. It may continue in this position until the beryl occupies the entire area near the smaller end.

A number of pieces of this intergrowth found on the dumps were massive and without any indication of possessing an external outline. The beryl plates in these are often distorted but show clearly that they are

often bounded by crystal forms, usually the prisms. Such intergrowths may represent the larger ends of large crystals, however, the top parts of crystals corresponding in size were not observed. It is believed, therefore, that these specimens may represent intergrowths of such a nature that the growth of the beryl was so completely disorganized by the simultaneous growth of the albite that it could not coalesce sufficiently to develop even the rough irregular skeleton crystals.

While the many parts and projections of the beryl contributing to the make-up of these irregular masses and tapered crystals are usually in parallel growth and probably connected, this is not always the case for small beryl crystals were observed that stood at an appreciable angle to the adjoining beryl. The small crystals were not tapered and had not reached the stage of development where the albite caused serious morphological interference with their growth.

The intergrowths are frequently associated with an adjoining graphic structure consisting of albite and a faint smoky variety of quartz. The surrounding albite may be of medium-grain or of the bladed variety, cleavelandite, which radiates from the beryl-albite intergrowths or other minerals preceding it from the same magma fraction. This radial habit of cleavelandite is often very important in establishing its relationship to the surrounding minerals—a feature that seems to have been overlooked or ignored by the advocates of the replacement hypothesis.

The minerals associated with the beryl-albite intergrowths are chiefly quartz, tourmaline, muscovite, apatite and garnet. These in every respect appear to be similar to those in the adjacent albite. There seems to be no particular reason why these minerals should not be the same within and outside of the beryl-albite whether they represent a replacement pair or are of contemporaneous origin. Hence they have no important diagnostic value.

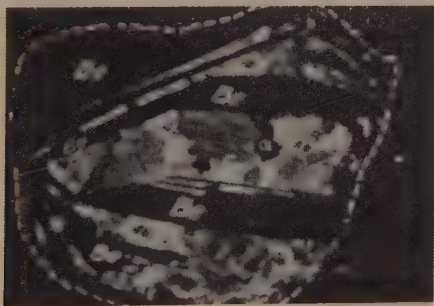


FIG. 3. Section parallel to and $\frac{1}{2}$ inch from that of Fig. 2 and shows the quick change in structure. Beryl (Be), Albite (Ab), Quartz (Q). $X=1.5$.

Microscopic examination shows that the albite within the beryl is comparatively fine to medium-grained and is not cross-cut by the narrow plates or blades of beryl as one would certainly find of frequent occurrence if the beryl replaced the albite, especially under the conditions illustrated in Figs. 2 and 3, where the blades of beryl are much narrower than the average width of the albite grains. At the extremities of these blades one occasionally finds grains of albite *growing around* the edge of the blade. This position of the beryl apparently "working into" the albite may lead one to conclude that the beryl replaces the latter, but such a conclusion based on this relationship must be erroneous for with *very few* exceptions the albite grains are not cross-cut by the beryl. The few exceptions that were noted undoubtedly represent sections cut close to an end or irregular edge of the beryl. A section along the line X—X' in Fig. 4 would show a cross-cutting relationship, however, below this extreme end no other section would show this feature. Hence, the very infrequent occurrence of cross-cutting mitigates against a hypothesis postulating the replacement of the albite by the beryl. Instead, it shows that occasionally an albite grain *grew around* the edge of the beryl when the growth of the latter was arrested.

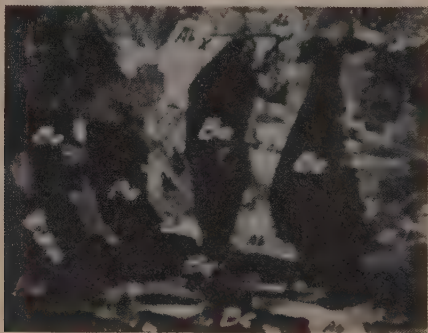


FIG. 4. Section parallel to the c axis and from the same crystal as Figs. 2 and 3. It shows the quick change in structure along the c axis. Beryl (Be), Albite (Ab). X=2.64.

The very long straight and sharp contacts between the beryl and albite as shown in Figs. 2 and 3 are especially noteworthy. Contacts of this kind are not favorable criteria of replacement. When they occur with other features adverse to replacement hypotheses they cannot be called exceptions to the rule of replacement. Sharp, linear contacts are very common between contemporaneous minerals when one of them possesses a strong tendency to form euhedral crystals. In these figures the blades are usually parallel to either the first or second order prisms.

The occurrence and relationship of these intergrowths seem to possess many features incompatible with any reasonable replacement hypothesis and one may raise the following questions concerning the replacement of the albite by the beryl or vice versa:

1. If the beryl was introduced in later solutions—

- (a) Why should it develop these unusual ramifications in this comparatively uniform material (albite with minor accessories) when it possesses a very strong habit of forming well-shaped euhedral crystals?
- (b) Why should the resulting crystals be so different at the opposite ends when beryl is a holohedral mineral?
- (c) Why do not the slender beryl blades consistently cut across the larger albite grains?

2. If the albite replaces the beryl—

- (a) Why does it replace one end of the crystal externally and the other end internally in order to develop tapered crystals with a core or irregular intergrowth of beryl and albite at the one end and be usually free from albite at the other?
- (b) Why does not the cleavelandite grow *into* the beryl instead of *away* from it as shown in Fig. 1?
- (c) Why should the albite follow directions parallel to the prism faces when the mineral does not have a cleavage or other structure that would influence replacement in these directions?
- (d) If the blade-like parts of the beryl are residua of replacement they would show rough pitted solution surfaces instead of being comparatively smooth and having a luster of the same quality as the external prism faces.

There appears, therefore, too many inconsistencies for one to accept a replacement hypothesis to account for the origin of this intergrowth of beryl and albite, that is, for either a replacement of the albite by the beryl or the replacement of the beryl by albite.

A contemporaneous origin for these two minerals is one which will permit a logical explanation for the various occurrences and characters shown by the specimens developed in these mines, and no doubt will explain the origin of those from other localities. Intergrowths of this kind are common among other pairs of minerals and, prior to the wave of replacement hypotheses during the past few decades, have been considered as originating from the crystallization of a magma fraction consisting essentially of the constituents involved in the intergrowths. In this instance the intergrowths may readily develop from a magma fraction rich in albite and beryl and produced as a late fraction of the

original pegmatite magma. A progressively contemporaneous crystallization of these two minerals may readily develop skeletonized beryl crystals which were simultaneously filled in by albite and the accessory minerals of this pegmatite magma fraction. The stronger crystal forming habit of beryl apparently gave this mineral the ability to control the structure and no doubt it slightly preceded the albite which offered an interference of such a nature that they together developed the tapered beryl-albite crystals and also the irregular intergrown masses of these minerals without any external form. In order for the massive intergrowths to develop both the beryl and albite must, of course, reach the point of crystallization at approximately the same time and the rate of crystallization of both must be sufficiently balanced in all directions until the beryl is exhausted or until its solution and solid phases are isolated by the vastly more abundant albite surrounding the latter. Under other, yet very similar, conditions and probably nearby, the intergrowth from its inception was irregular and unbalanced, thereby offering relatively greater advantages for one or the other of the crystallizing substances to predominate in its development in some particular direction depending upon the physico-chemical conditions present. The much larger quantity of albite always present in these solutions caused the direction favoring this mineral to expand while at the same time in the opposite direction, as the beryl is gradually excluding the albite and becoming lower in concentration, the intergrowth develops a reduced cross-sectional area. This reduction of area may be somewhat influenced by the external crowding of the albite. Since the tapered end of the beryl crystal is always clearer and often of gem variety, it indicates that this part must have developed more leisurely when the concentration and supersaturation of the beryl was very low.

Under nearly similar physico-chemical conditions the beryl may reach an advanced stage of crystallization before the albite became supersaturated sufficiently for it to crystallize, and consequently the beryl will develop euhedral crystals unless adjoining substances should interfere. The albite (cleavelandite) being the next to crystallize would attach itself to the exposed parts of the beryl and develop a crust. The cleavelandite may be followed by quartz which is frequently the last mineral to crystallize in quantity. It appears to the writer that the material represented in Hess' Fig. 6² belongs in this category and does not indicate a series of replacements as he described.

² Hess, F. L., The natural history of pegmatites: *Eng. & Min. Jour. Press*, vol. 120, pp. 289-298, 1925.

SUMMARY

The beryl-albite intergrowths occurring at the Golding-Keene and the Strickland feldspar mines are shown to be of such a nature that an origin through the processes of replacement is most unlikely, while on the other hand their relationship and other features clearly indicates a contemporaneous crystallization.

HILGARDITE, A NEW MINERAL SPECIES, FROM CHOCTAW SALT DOME, LOUISIANA

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ABSTRACT

In the insoluble residue from a brine well in the Choctaw Salt Dome, Louisiana, is found an unusual mineral assemblage. Hilgardite, a new species, is found there in association with anhydrite, danburite, boracite, and other minerals. Hilgardite is monoclinic-domatic; $a:b:c=1.0147:1:0.5585$, $\beta=90^{\circ}00'$. It is clear and colorless with a vitreous luster. Cleavage: $\{010\}$ perfect, $\{100\}$ difficult. $H=5$. $G=2.71$. Biaxial positive, $2V=35^{\circ}$, $r>v$, $nX=1.630$, $nY=1.636$, $nZ=1.664$. The dimensions of the unit cell: $a_0=11.35\text{\AA}$, $b_0=11.12\text{\AA}$, $c_0=6.20\text{\AA}$. The space group is either Pc or Pm . The chemical formula expressing the content of the unit cell may be written: $\text{Ca}_8(\text{B}_6\text{O}_{11})_3\text{Cl}_4 \cdot 4\text{H}_2\text{O}$. Named in memory of E. W. Hilgard.

INTRODUCTION

Studies of the water-insoluble residues of rock salt from Gulf Coast salt plugs reveal the presence of an interesting and rather distinctive mineral assemblage. Residues from the different salt plugs have a similar composition,¹ but the one from the salt plug of Choctaw Salt Dome, Iberville Parish, Louisiana, contains several unusual minerals which have not been found in the others. Hilgardite, the new mineral species described in this paper, danburite, boracite, and magnesite were found only in the Choctaw residue.

The Choctaw residue comes from the Solvay Process Company number four brine well and represents water-insoluble materials liberated from the rock salt during the production of brine. The insoluble material accumulated at the bottom of the well and was brought to the surface when the well was washed. The samples studied were collected from the sump into which the washings were discharged. As the casing through which the brine is pumped extends to some 1200 feet below the top of the salt, this residue comes from well within the salt plug. Analyses of salt cores from a test-well drilled close to the number four well indicate that the rock salt contains from two to four per cent of insolubles. The brine from number four well shows a trace of potash salts, and its production is accompanied by the liberation of appreciable amounts of methane, hydrogen sulphide and other gases.

The residue is a light bluish-gray sand of fine-to-medium-sand size and is composed chiefly of anhydrite grains. Fragments of friable, gray,

¹ A general discussion of the residues will be presented in "Water-Insoluble Residues from the Rock Salt of Louisiana Salt Plugs," which the junior author will publish in the *Bulletin of the American Association of Petroleum Geologists* for October, 1937.

argillaceous sandstone, and of black, calcareous shale are also present. The sandstone is composed of poorly sorted quartz sand and a subordinate amount of the heavy minerals tourmaline, zircon, rutile, ilmenite and leucoxene. The minerals² which have been identified from the residue, exclusive of those in the sandstone, are, in order of abundance: anhydrite, dolomite, magnesite, hilgardite, pyrite, calcite, boracite, danburite, quartz, sulphur, limonite, hematite, marcasite, hauerite, and gypsum.

MORPHOLOGY

Hilgardite, the new species, is present in the residue as distinct and well-formed crystals. It belongs to the domatic class of the monoclinic system, and is thus unusual in that only one other previously described mineral, clinohedrite, falls in this class. The goniometric and x -ray measurements both give rectangular axes; but, since the external morphology shows only a plane of symmetry, monoclinic symmetry is indicated with $\beta = 90^\circ 00'$. Most of the crystals are similar in appearance with a pronounced tabular habit and hemimorphic aspect as shown in Figs. 1, 3 and 4. They range in size from 0.5 mm. to 15.0 mm. in maximum dimension.



FIG. 1. Crystals of hilgardite lying on (010)
(Natural size)

² A more detailed paper on the paragenesis and crystallography of the minerals associated with hilgardite is in preparation.

TABLE 2. HILGARDITE: ANGLE-TABLE

Hilgardite— $\text{Ca}_8(\text{B}_6\text{O}_{11})_3\text{Cl}_4 \cdot 4\text{H}_2\text{O}$ Monoclinic; domatic— m

$$a:b:c = 1.0147:1:0.5585$$

$$\beta = 90^\circ 00'$$

$$p_0:q_0:r_0 = 0.5504:0.5585:1$$

$$\mu = 90^\circ 00'$$

$$r_2:p_2:q_2 = 1.7905:0.9855:1$$

$$p_0' = 0.5504$$

$$q_0' = 0.5585$$

$$x_0' = 0.00$$

Forms	Calculated				
	ϕ	$\rho = C$	ϕ_2	$\rho_2 = B$	A
<i>b</i> 010	0°00'	90°00'	—	0°00'	90°00'
<i>K</i> $\bar{1}$ 30	—18 11	90 00	180°00'	161 49	108 11
<i>L</i> $\bar{2}$ 30	—33 18½	90 00	180 00	146 41½	123 18½
<i>M</i> $\bar{1}$ 10	—44 35	90 00	180 00	135 25	134 35
<i>E</i> 0 $\bar{1}$ $\bar{1}$	180 00	150 49	—90 00	119 11	90 00
<i>p</i> 111	44 35	38 06	61 10½	63 55½	64 20
<i>Q</i> 1 $\bar{1}$ $\bar{1}$	135 25	141 54	—61 10½	116 04½	64 20
<i>q</i> $\bar{1}$ 11	—44 35	38 06	118 49½	63 55½	115 40
<i>R</i> 2 $\bar{1}$ $\bar{1}$	116 54	129 00½	—42 15	110 35	46 08
<i>r</i> $\bar{2}$ 11	—63 06	50 59½	137 45	69 25	133 52
<i>s</i> $\bar{3}$ 11	—71 18½	60 10½	148 48	73 52	145 15

PHYSICAL AND OPTICAL PROPERTIES

The cleavage of hilgardite is perfect and easy parallel to {010} and just as good but difficult parallel to {100}. The hardness is 5. The specific gravity determined by suspension in bromoform is 2.71. On fresh surfaces it is clear and colorless with a vitreous luster. The optical properties of hilgardite are as follows:

	$n(\text{Na})$	Positive
X : $c = 88\frac{1}{2}^\circ$	1.630	±0.002
Y : $b[010]$	1.636	
Z : $c = 1\frac{1}{2}^\circ$	1.664	
		2V = 35°
		$r > v$

X-RAY MEASUREMENTS

Weissenberg photographs were taken of the zero and first layer lines with both $b[010]$ and $c[001]$ as the axes of rotation. These photographs gave an apparent orthorhombic net with the dimensions:

$$a_0 = 11.35 \text{ \AA}, b_0 = 11.12 \text{ \AA}, c_0 = 6.20 \text{ \AA}$$

giving $a_0:b_0:c_0 = 1.027:1:0.559$. This ratio compares favorably with the morphological ratio: $a:b:c = 1.0134:1:0.5585$, but inasmuch as only poor crystals were available for goniometric measurement it is believed

that the x -ray data are more reliable. The density of hilgardite is 2.71, the volume of the unit cell 786 cubic Å, and hence the molecular weight of the unit cell $M_0 = 1290$.

Examination of the Weissenberg photographs showed certain spots to be missing that should be present in true orthorhombic symmetry. Accordingly, Laue photographs were taken normal to the {010} and {100} cleavages. The only symmetry element shown in the former is a two-fold axis, and in the latter only one reflection plane. The monoclinic character of hilgardite is thus definitely established even though the angle β is $90^\circ 00'$ and extinction angle is negligible.

Hilgardite falls in the space group $C_s^1 - Pc$ or $C_s^2 - Pm$ as determined by the space group criteria:

$h00$	halved
$0k0$	halved
$00l$	halved
$h0l$	present for h odd
hkl	present for h odd
hkl	present for $h+l$ even

COMPOSITION

Table 3 gives the chemical analysis and the atomic content of the unit cell using the molecular weight, $M = 1290$.

TABLE 3. ANALYSIS OF HILGARDITE AND CONTENT OF UNIT CELL

	1	2	3	4	5
CaO	34.41	35.14	.6265	Ca 8.1	CaO 35.67
B ₂ O ₃	49.18	50.22	.7211	B 18.6	B ₂ O ₃ 49.86
H ₂ O	6.31	6.44	.3580	H ₂ 4.6	H ₂ O 5.73
Cl	10.37	10.59	.2986	Cl 3.9	Cl 11.28
Insol.	1.89				
	102.16	102.39			102.54
Less O	2.34	2.39			2.54
	99.82	100.00			100.00

1. Analysis by F. A. Gonyer. 2. Insolubles deducted. 3. Molecular ratio. 4. Atomic contents of unit cell. 5. Calculated composition for $\text{Ca}_8\text{B}_{18}\text{O}_{33}\text{Cl}_4 \cdot 4\text{H}_2\text{O}$.

Hilgardite belongs to the group of soluble borates, but the presence of chlorine makes it distinct from the others. A comparison of the chemical formulae below shows the relation that it bears to hydroborocite and colemanite.

Hydroboracite	$\text{MgCaB}_6\text{O}_{11} \cdot 6\text{H}_2\text{O}$
Colemanite	$\text{Ca}_2\text{B}_6\text{O}_{11} \cdot 5\text{H}_2\text{O}$
Hilgardite	$\text{Ca}_8(\text{B}_6\text{O}_{11})_3 \cdot \text{Cl}_4 \cdot 4\text{H}_2\text{O}$

Pyrognostics. On charcoal before the blowpipe hilgardite fuses at 2 to a compact white globule, and colors the flame the yellowish green of boron. In the closed tube, water is driven off easily that gives a strong acid reaction. At a low temperature some chlorine is apparently driven off which dissolves in the water.

It seems fitting to name this new borate found in a salt well in Louisiana, hilgardite, after E. W. Hilgard, an early geologist of Mississippi and one of the first to make observations on the salines of Louisiana.

ACKNOWLEDGMENTS

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MASSIVE LOW-FLUORINE TOPAZ FROM THE BREWER MINE, SOUTH CAROLINA*

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INTRODUCTION

A mass of fine-grained topaz, unlike any deposit heretofore described, because of its large size and low-fluorine content, forms a part of the gold-bearing lode at the Brewer Mine near Jefferson, Chesterfield County, South Carolina.

The Brewer gold mine is one of the early discoveries of the Southern Appalachian region. In the course of intermittent mining operations during the last 100 years a superficial placer deposit has been largely stripped from an area of 20 or 30 acres and numerous pits have been excavated, one of them 300 feet across and 140 feet deep.

The country rock is a quartz-sericite schist that locally preserves the structure of a fine-grained waterlaid tuff, probably a rhyolite, from which it was derived. Certain layers of it carry much pyrophyllite. Exposures of a granite that intrudes the schist appear on the north and west at distances of a mile to a mile and a half.

The lode extends nearly half a mile along a broad ridge about 200 feet high between Little Fork Creek and Lynch's River. It is a large body composed chiefly of fine-grained quartz that shows the pattern of a breccia that it has replaced. As exposed in the largest excavation, the Brewer pit, the lode is generally weathered to depths ranging from 40 to 60 feet. At greater depths the rock is firm and flint-like. Generally it contains from 2 to 5 per cent of pyrite, unevenly distributed as aggregates and individual crystals. Small grains of enargite are sparingly scattered through parts of the rock and the following minerals not seen by the authors are reported: covellite,² cassiterite,³ and bismuth ochre (bismite) and native bismuth.⁴ Assays of samples from the Brewer pit show from .045 ounce to .13 ounce of gold per ton. Spectrographic tests of these samples by George Steiger showed no platinum or palladium.

In the weathered zone much of the flinty appearing quartz rock is disintegrated to a very fine white sand, some of it snow-white and some

* Published by permission of the Director, U. S. Geological Survey.

¹ Field work and geology by Pardee; microscopic work by Glass; and analytical chemical work by Stevens.

² Becker, G. F., Gold fields of the southern Appalachians: *U. S. Geol. Survey, 16th Ann. Rept.*, pt. 3, p. 279, 1895.

³ Clark and Chatard, *Am. Jour. Sci.*, 3d ser., vol. 28, p. 25, 1884.

⁴ Tuomey, M., *Geology of South Carolina*, p. 97, 1848.

more or less iron-stained. Alongside the sandy material are bodies with the structure of a breccia cemented with iron oxides. Both kinds carry fine particles of free gold. In several places the lode quartz is cut by veins of coarse white quartz ("bull quartz") that show no other minerals except rare groups of ilmenite plates.

As mentioned farther on, the quartz rock contains scattered grains of topaz. This fact was suspected by Graton,⁵ who examined the deposit some 30 years ago. He mentions the quartz as "penetrated by numerous prismatic crystals, apparently of secondary origin, possibly topaz."

MASSIVE TOPAZ

Physical and optical characteristics

A rock composed almost exclusively of very fine-grained topaz forms a considerable part of the lode northwest of the Brewer pit. Its exposures in place occupy an area about 25 feet wide and 50 feet or more long, but a greater extent of the body is indicated by the distribution of "float" on the slope above the outcrop. Loose fragments about the size of half a brick, some of them waterworn, are abundant in the waste heaps of placer pits along the slope below. In general appearance the topaz resembles the undecomposed quartz rock of the lode. In fact the two look so much alike that in a cursory examination they are not likely to be distinguished from one another unless the relatively high gravity (3.5) of the topaz aggregate should be noticed. Some specimens show the pattern of a breccia and others are marked with wavy lines or bands representing structures of a rock that the topaz has replaced. Contact relations of the two indicate that the topaz is later than the quartz rock.

The topaz mass is not described as such in previous reports of the Brewer Mine. Apparently it has been classified heretofore along with the siliceous body as "flinty quartz." Its texture is very similar to that of flint or chert. It has a waxy luster, and its hardness is about 7, less than for normal topaz. It breaks with a conchoidal fracture and separates along joints into rather small smooth-faced blocks, some of which have forms suggesting pyramids and wedges. The color of these blocks of topaz is gray, mottled or streaked with white cloud-like bands in a dull gray sub-translucent mass. Some of the weathered fragments show the red staining of iron oxide. Some fragments which are gray on the surface are deep carnelian red on the interior, the iron stain being hidden by a gray-white leached zone a few millimeters thick. The iron stain appears to be due to the oxidation of small inclusions of pyrite. As shown by

⁵ Graton, L. C., Reconnaissance of some gold and tin deposits of the southern Apalachians: *U. S. Geol. Survey, Bull.* 293, pp. 90, 91, 1906.

the chemical analysis, the actual amount of iron present is small. A sample yielded .01 ounce of gold per ton by assay.

Thin sections show that the exceedingly fine grains of the massive topaz vary slightly in size among themselves. They are only a few microns in diameter and closely resemble the granules in a very fine-grained chert. The granules are arranged throughout the mass in irregular alternating bands of coarser and finer texture, thus giving a streaked appearance to the surface pattern of the groundmass. In transmitted light the thin section surface shows a stippled or shagreen effect, again in this way resembling chert.

On account of its fine-grained character only the mean index of refraction could be determined on the massive topaz, this is $n=1.631$, which corresponds to β in the crystals of the more coarsely granular variety associated with it, and thus indicates the same chemical composition.

Chemical Analysis

For the determination of silica and fluorine a sample weighing 0.2141 g. was fused with 5 g. sodium carbonate and the resulting cake thoroughly leached with water. The residue was ignited at a low heat, again fused with 5 g. sodium carbonate, the cake leached with water, and the second extract added to the first.

From the combined filtrates, which contained all of the fluorine, silica was removed by two precipitations with zinc oxide, as recommended by Hoffman and Lundell.⁶ The residue, left after leaching the sodium carbonate cake with water, and the zinc precipitates were combined for the determination of silica by dehydration in the usual way.

The extracted fluorine was determined nephelometrically.⁷ The solution, free from silica, was made just acid to methyl red and diluted to 200 ml. in a volumetric flask, giving a sodium chloride concentration of 55 grams per liter. From a 25 ml. aliquot calcium fluoride was precipitated as a colloid by reducing the solubility with alcohol and stabilizing the suspension with gelatin. At the same time 25 ml. of a standard sodium fluoride solution (0.2000 g. NaF and 55 g. NaCl per liter) was precipitated colloiddally in the same way and the two opalescent solutions compared in a nephelometer to give a preliminary reading. In accordance with this estimate the unknown solution was diluted to within 5 per cent of equality with the standard in fluoride concentration, adding sodium

⁶ Hoffman, J. I., and Lundell, G. E. F., Determination of fluorine and of silica in glasses and enamels containing fluorine: *Bur. Standards Jour. Research*, vol. 3, p. 581, 1930.

⁷ Stevens, R. E., Nephelometric determination of fluorine: *Ind. and Eng. Chem., Anal. Ed.*, vol. 8, p. 248, 1936.

chloride to keep its concentration 55 g. per liter. Aliquots of the new unknown solution and of the standard were then precipitated colloiddally and compared in the nephelometer to give a final reading.

Water was determined by the Penfield method on a sample ground to an impalpable powder, using sodium tungstate as a flux, and heating intensely with a full blast. Alumina was determined in the usual way on a separate sample.

The analysis of the massive topaz is as follows:

SiO ₂	33.00
Al ₂ O ₃	56.76
Fe ₂ O ₃	trace
H ₂ O—	0.04
H ₂ O+	2.67
F	13.23
	<hr/>
	105.70
Minus O=F	5.57
	<hr/>
	100.13

The analysis is confirmed by the low specific gravity and by the optical properties.

From a review of the literature, it appears that the Brewer material shows the lowest F content and the highest H₂O content of any topaz yet described.

Three determinations of the specific gravity of a lump of the massive topaz were made by the water immersion method, correcting the results for temperature. The average of these gave a specific gravity of 3.509 ± 0.001 referred to water at 25°C., or a density of 3.499.

This specific gravity is the lowest recorded for topaz, as far as the writers know, and it accords with the low fluorine and a high hydroxyl content.

OTHER TYPES OF TOPAZ IN THE BREWER MINE

White granular topaz aggregate

In addition to the massive fine-grained topaz described, the Brewer lode contains disseminated fine grains and, in one place at least, an aggregate of coarser grains. Specimens from a boulder found in an old placer working (Tanyard pit) about 1,000 feet south of the Brewer pit, are aggregates of white granular topaz from which some other more soluble mineral or minerals have been dissolved. The mass has a sponge-like structure, some of the cavities being rounded, others angular. The aggregate consists of clear, clean, colorless grains, about .01 mm. to .5

mm. in diameter (the prismatic grains being slightly longer than wide), loosely held together merely by interlocking. Apparently all cementing materials have disappeared and the mass is friable enough to be crushed with the hands. A few spots of iron oxide suggest that probably pyrite at one time occupied at least some of the spaces. If the ore minerals were removed from a specimen photographed and described by Singewald and Milton⁸ in their paper on "Greisen and associated mineralization at Silver Mine, Missouri," the remaining vesicular topaz mass would resemble the granular topaz aggregate from the Brewer Mine.

Optical properties

Generally the grains of the white granular aggregate show, in thin section, a rounded outline; only few give a feeble suggestion of euhedral form. A considerable number of the larger grains show a rounded or elongated nucleus of topaz of different orientation from the surrounding material. Commonly a basal section of such a nucleus gives a small optical axial angle which indicates that it, like the surrounding topaz, has a low-fluorine, high-water content. A few minute inclusions of some birefracting mineral with low index, probably quartz, are observed, but the gas or liquid bubble inclusions often seen in topaz are absent in the specimens examined. A very few specks of a dark yellow-brown, high-index mineral are also present. The amount of impurities, however, is trifling and the loose granular aggregate is essentially pure topaz.

The individual topaz grains are colorless and transparent, most of them are rounded, but some are slightly prismatic. Grains showing basal cleavage are numerous. Their optical axial angle, $+2V=45^{\circ}$ to 48° , is unusually small for topaz. The dispersion is distinct, $r > v$. Indices of refraction measured in white light are found to be: $\alpha=1.629$, $\beta=1.631$, $\gamma=1.638$. $B=0.009$. The optical properties were determined with index liquids standardized at the temperature of the room at the time the measurements were made and are correct to ± 0.0005 .

The small optical axial angle and high indices of refraction indicate the material to be of the same low fluorine (13.23%) and high water (2.67%) content as the massive topaz. (Constant relation between fluorine-water content and optical properties are discussed further on.)

DISSEMINATED TOPAZ

In thin section under the microscope a specimen of the unweathered lode rock from the Brewer pit is found to contain a few small scattered

⁸ Singewald, J. T., Jr., and Milton, Charles, Greisen and associated mineralization at Silver Mine, Missouri: *Econ. Geol.*, vol. 24, no. 6, p. 574, September, 1929. (Fig. 4, p. 574.)

grains of topaz, otherwise it consists of quartz and pyrite, with a very insignificant amount of sericite. Optically the disseminated topaz is the same as the others already described.

The groundmass of the lode rock is composed chiefly of very small anhedral grains, but it includes aggregates of coarser grains of quartz. A few of these larger quartz grains contain rounded islands or nuclei of quartz, apparently remnants of pre-existing grains enlarged by the later addition of quartz. Pyrite is abundant as euhedral crystals ranging from dust-like specks to individuals 1.5 mm. in diameter. Sericite forms tiny flakes interstitial in the quartz aggregate. It may replace the topaz or quartz.

Some of the specimens from the zone of weathering consist only of quartz and sericite. Samples of sand taken from disintegrated rock in place on the south side of the Brewer pit showed a range in composition approximately as follows: No. 1, topaz 0, quartz 100 per cent; No. 2, topaz 5 per cent, quartz 95 per cent; No. 3, topaz 15 per cent, quartz 85 per cent. The boulder of white granular topaz already described is a friable mass of topaz grains almost entirely without quartz. The variation thus shown by the few samples that have been studied suggests that adequate sampling would reveal a complete gradation from pure quartz to pure topaz.

The uniformity in optical properties of topaz from different parts of the Brewer Mine signifies uniformity in chemical composition, which in turn suggests a common origin. The area seems to have been saturated by magmatic solutions which were rich in fluorine, silica, and sulphides, and deficient in alkalis.

CONSTANT RELATION BETWEEN THE FLUORINE-WATER CONTENT AND THE OPTICAL AND PHYSICAL PROPERTIES OF TOPAZ

According to Penfield⁹ the variation which topaz shows both in chemical composition and physical properties results from an isomorphous replacement of fluorine by hydroxyl. As the hydroxyl replaces fluorine the value for $2E(2V)$ decreases. This relation is so constant that the percentage of H_2O can be estimated from the value of $2E(2V)$. Likewise the indices of refraction show a progressive change along with the variation in $2E(2V)$. As the OH replaces F the indices of refraction increase and the strength of the double refraction decreases as shown by the following extremes recorded in the literature:

⁹ Penfield, S. L., and Minor, J. C., Jr., On the chemical composition and related physical properties of topaz: *Am. Jour. Sci.*, 3d ser., vol. 47, p. 387, 1894.

	Utah	Minas Geraes	Brewer
F	{ 20.33	{ 15.48	{ 13.23
H ₂ O	{ 0.19	{ 2.45	{ 2.67
2V	67°	49°	48°±
α	1.6070	1.6294	1.629
β	1.6100	1.6308	1.631
γ	1.6180	1.6375	1.638
B	.0110	.0081	.009 all±.0005.
G	3.565	3.532	3.509

Optical properties on a crystal of rose topaz from the Urals noted by Sabot¹⁰ are: $+2V = 49^\circ 22'$; $\alpha = 1.6293$, $\beta = 1.6308$, $\gamma = 1.6379$. No chemical analysis was made of this topaz crystal. The indices, when rounded off to three decimal places, agree exactly with the Brewer topaz.

SUMMARY

Topaz of a fine-grained chert-like character forms a large part of the gold-bearing lode at the Brewer mine, Chesterfield County, South Carolina. The enormous amount of this massive topaz, together with its unusual fine-grained texture and its low-fluorine content, constitutes the most unique and the most noteworthy occurrence of such a deposit ever to be recorded.

The massive topaz has a fine-grained quartz-like appearance in hand specimen. Microscopically the texture closely resembles chert. The mean index of refraction is $n = 1.631$, which corresponds to the low fluorine and high water contents. The chemical analysis shows fluorine 13.23 per cent, the lowest fluorine content, and water 2.67 per cent, the highest water content on record.

Granular topaz aggregates found in the associated rocks furnish the best material for optical observations. The small, clean, euhedral grains give complete optical data that substantiates the chemical analysis. The $+2V$ angle $= 48^\circ$; the indices of refraction are: $\alpha = 1.629$, $\beta = 1.631$, $\gamma = 1.638$. $B = 0.009$.

Uniformity of the optical data of the massive, granular, and disseminated topaz indicates uniformity of chemical composition and suggests a common origin. Previously existing rocks of the area apparently reacted with solutions rich in fluorine, silica, and sulphides, and deficient in alkalis, and became replaced by topaz and quartz, and small quantities of sulphides.

¹⁰ Sabot, R. C., *Thèse* 519, U. Genève, 1914; *Zeits. Krist.*, vol. 56, p. 631, 1922.

NOTES AND NEWS

TELLURIUM MINERALS OF NEW MEXICO

WM. P. CRAWFORD, *Bisbee, Arizona.*

Petzite was reported from the Red River District, Taos County, in 1904, and is the first recorded occurrence of a tellurium mineral in New Mexico. Tetradymite was identified in 1908 at Sylvanite and since then tellurium minerals have been described from other localities. Unfortunately for collectors, many of the localities are now exhausted and none are noted for exceptionally fine mineral specimens.

A total of ten tellurium minerals have been reported (Fig. 1); eight have been reliably identified and two, sylvanite and calaverite, are probably mixtures of a telluride and native gold.

MINERAL	COUNTY
Tellurium	Catron
Tetradymite	Colfax, Grant, Dona Ana, Hidalgo, Sierra
Copper telluride	Dona Ana
Altaite	Dona Ana
Hessite	Hidalgo, Dona Ana,* Sierra*
Petzite	Taos, Dona Ana*, Sierra*
Sylvanite	Hidalgo*, Sierra*
Calaverite	Sierra*
Montanaite	Dona Ana
Durdenite	Catron

* Indicates identity is doubtful.

CATRON COUNTY

Ballmer¹ identified native tellurium in vein material collected at a gold prospect in southern Catron County, 41 miles northwest of Silver City. The tellurium was associated with bismuthinite and pyrite in a gangue composed principally of quartz and fluorite. The tetradymite reported from this locality proved a mixture of tellurium and bismuthinite. Durdenite² was identified in several oxidized specimens and is a new and unreported occurrence of this mineral. The exploitation of the deposit as a source of tellurium was unsuccessful but a number of mineral specimens were obtained.

COLFAX COUNTY

Tetradymite has been found in small amounts at the Aztec mine, Ute Creek District. A mine report (dated 1916) by E. H. Perry and Augustus Locke mentioned chlorite carrying "scattered grains of pyrite and native

¹ Ballmer, G. J., Native tellurium from northwest of Silver City, New Mexico: *Am. Mineral.*, vol. 17, pp. 491-492, 1932.

² Ballmer, G. J., *personal communication.*

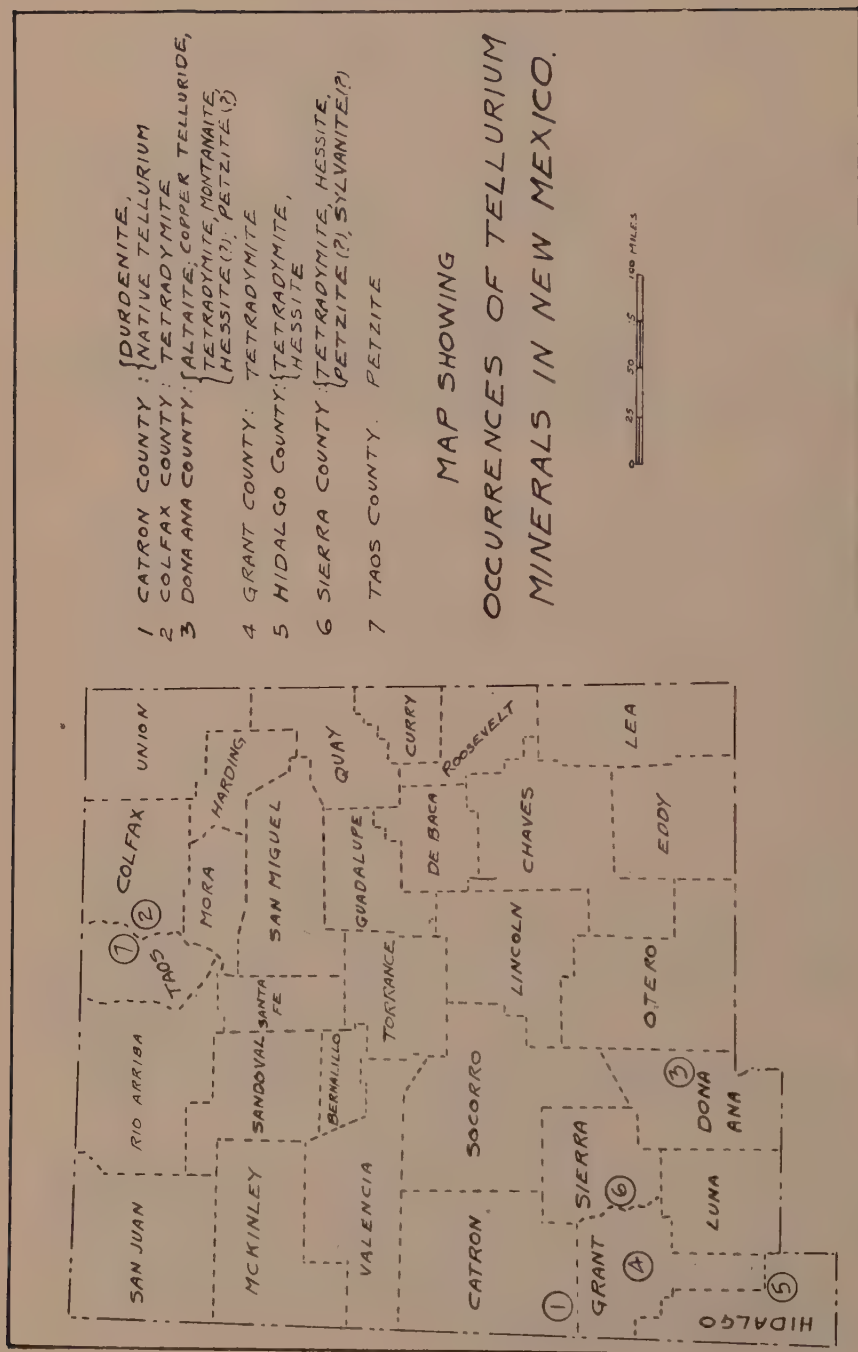


Fig. 1. Occurrences of Tellurium Minerals in New Mexico.

gold, with occasional gray metallic specks which may be telluride or selenide of gold." No attempt was made to identify the mineral.

A detailed study of the mine later made by Chase and Muir³ showed tetradymite but no telluride or selenide of gold. "Besides masses of coarse and fine native gold, this ore contained quartz, limonite, garnet, pyroxene, and other silicates, a little pyrite and chalcopyrite, and always an appreciable amount of bismuth telluride. The latter mineral, tetradymite, was identified by A. J. Weinig. Native gold in coarse particles was embedded in it, yet practically uncombined with tellurium."

DONA ANA COUNTY

Tetradymite has been identified in the ores of the Ben Nevis, Crested Butte, Texas Main, and Memphis mines in the Organ District. Oxidized tetradymite from the South Shaft of the Memphis mine exhibited, in polished surfaces, a graphic pattern which Dunham⁴ attributes to a possible intergrowth of pure tetradymite with one rich in sulphur or selenium. A similar structure is shown in bismuth telluride from the Gold Bug mine, Bannack mining district, Montana, when it is etched with nitric acid.⁵

Altaite is found at the Hilltop mine in association with pyrite and galena. It yields in the oxidation zone a very soft mineral which is probably a lead tellurite.⁶ This oxidation product of altaite from the Hilltop mine, while mentioned by Schneiderhöhn and Ramdohr,⁷ has not been found in sufficient quantity for analysis and has not been named.

Hessite and petzite: the identity of these tellurides in Dona Ana County is based upon qualitative tests for tellurium made by L. B. Bentley on hand specimens in 1914. None of the material is now available for microscopic study. Hessite was reported from the Little Buck mine, as rich silver ore gave strong tellurium tests. Gold-silver ore from the Gonzales prospect contained tellurium and it may be in the form of hessite or petzite.

Copper telluride: hand specimens from the Eureka and Rickardite claims, Organ District, contained small grains of a mineral which gave strong qualitative tests for copper and tellurium, and which, on the

³ Chase, Chas. A., and Muir, Douglas, The Aztec Mine, Baldy, New Mexico: *Trans. Am. Inst. Min. Eng.*, vol. 69, p. 276, 1923.

⁴ Dunham, K. C., The geology of the Organ Mts. New Mexico: *N. M. Bur. of Mines and Min. Res., Bull.* 11, p. 132, 1935.

⁵ Shenon, P. J., Geology and ore deposits of Bannack and Argenta, Montana: *Montana Bur. of Mines and Geology, Bull.* 6, p. 54, 1931.

⁶ Dunham, K. C., *op. cit.*, p. 159.

⁷ Schneiderhöhn, H., and Ramdohr, P., *Lehrbuch der Erzmikroskopie*. Bd. II, pp. 264-265, Berlin, 1931.

basis of these tests, was identified as rickardite by L. B. Bentley. Two specimens in Mr. Bentley's collection were examined by the writer and it is his belief that the copper telluride is weissite rather than rickardite. It occurs as small grains in a calcite gangue and none of the material shows the characteristic purple color of rickardite. The color of a freshly scratched surface is a bluish-black, tarnishing to a deep black and resembles the type specimen of weissite from the Good Hope mine.⁸ Only a few specimens were recognized as copper telluride while the Eureka and Rickardite claims were being worked and none is available now.

Specimens of oxidized bismuth ore from the Memphis mine contained a yellow mineral giving positive micro-chemical tests for bismuth and tellurium. No unaltered tetradymite was found and identification was tentatively made as montanaite.⁹

GRANT COUNTY

A specimen of tellurium-bearing gold ore from a prospect in the Little Burro Mountains, given the writer by Mr. Ira L. Wright, was identified as tetradymite by Dr. G. M. Schwartz, University of Minnesota. It occurred in masses 2.5 cm. in greatest diameter, in association with chalcopyrite, malachite(?), and limonite. Large areas of the tetradymite are altered along contacts and cleavages, but show no intergrowth. Material from this property is no longer obtainable. This occurrence of tetradymite in Grant County has not been previously reported.

HIDALGO COUNTY

Tetradymite has been found in the Sylvanite mining district¹⁰ in the ores of the Hand Car, Golden Eagle, Gold Hill, Ridgewood, Little Mildred,¹¹ and Pearl claims. Native gold, uncombined with tellurium, is associated with the bismuth telluride.

Hessite has been observed in polished sections of tetradymite from the Little Mildred claim¹² and the two tellurides are intergrown and contemporaneous with native gold.

Sylvanite has been reported from the Sylvanite district but specimens examined by the writer proved a mixture of tetradymite and native gold.

⁸ Crawford, Wm. P., Weissite—A new mineral: *Am. Jour. Sci.*, vol. 13, pp. 345-346, 1927.

⁹ Dunham, K. C., *op. cit.*, p. 160.

¹⁰ Lindgren, Waldemar, Graton, L. C., and Gordon, Chas. H., The ore deposits of New Mexico: *U. S. Geol. Survey, Prof. Paper* 68, pp. 341-343, 1910.

Short, M. N., and Henderson, E. P., Tetradymite from Hachita, N. M.: *Am. Mineral.*, vol. 11, pp. 316-317, 1926.

¹¹ Crawford, Wm. P., and Johnson, Frank, *personal notes*.

¹² Lasky, S. G., *oral communication*.

SIERRA COUNTY

Small amounts of tetradymite and free gold were shown in vein material from a surface trench on the Copper Flat mining claims, Hillsboro mining district.¹³

Calaverite, petzite, and sylvanite were reported present in ores from the Lookout mine, Tierra Blanca district, but no investigation of the tellurides was made until 1931 when G. T. Harley collected a small suite of specimens from the Lookout. The only telluride identified by Mr. S. G. Lasky in this suite was hessite. Lasky found native gold, younger than the silver telluride, traversing the silver telluride in minute veinlets.¹⁴ The gold was present in varying amounts and perhaps explains the gold-silver tellurides reported in the earlier mined ores. The deposit is exhausted.

TAOS COUNTY

Petzite¹⁵ has been reported from several mines in the Red River District. It is said to have been present in siliceous ore from the Sampson mine and in some of the ore from the Memphis mine. Quartz boulders containing a mixture of pyrite and a telluride were found in the Independence mine in 1904, and is the first reported occurrence of a tellurium mineral in New Mexico. "Qualitative tests showed the presence of tellurium but the telluride, small lustrous grains of dark-gray color, was so intimately associated with the pyrite that a separation for analysis was too difficult." A specimen from the original find is now in the possession of Mr. Wm. Earle and shows grains of native gold included in the telluride. The mineral in this hand specimen resembles petzite but as Mr. Earle was unwilling to sell or loan the specimen for polishing, microscopic examination was impossible. Most collectors of tellurides will have a kindred feeling for Mr. Earle.

A NEW OCCURRENCE OF GYPSUM IN KENTUCKY

A. C. MUNYAN, *Lexington, Kentucky.*

Many small deposits of gypsum have long been known in Kentucky; some of the most notable being those found in the limestone caves of Edmonson County, and other caves of the western part of the State. Richardson¹ lists a few of the occurrences more widely known, such as

¹³ Harley, Geo. T., The geology and ore deposits of Sierra County, N. M.: *N. M. Bur. of Mines and Min. Res., Bull.* 10, p. 164, 1934.

¹⁴ Harley, G. T., *op. cit.*, p. 109.

¹⁵ Lindgren, Graton, and Gordon, *op. cit.*, pp. 87-88.

¹ Richardson, C. H., Mineralogy of Kentucky: *Ky. Geol. Sur., Ser.* 6, pp. 85-87, 1925.

the bedded deposits in the Niagaran shales on the eastern flank of the Cincinnati Arch, and the occasional crystals found in the cavities of the iron ores which occur in widely separated regions over the State.

The deposit under discussion is thought to be somewhat unique, although similar occurrences are known in other regions of the United States. In the early part of 1937, Mr. D. J. Jones, State Geologist of Kentucky, was asked to identify some crystals from the White City Mine of the Hart Coal Company near Morton's Gap in Hopkins County, Kentucky. The mineral proved to be selenite crystals of unusual development compared to those found elsewhere in Kentucky. Further investigation by Mr. Jones and the writer, with a visit to the mine, disclosed an abundance of this mineral in an air entry of the coal mine.

The Hart Coal Company is mining at present the No. 11 coal, in the White City Mine. A few feet above this coal lies the No. 12 seam, the interval between the two seams being occupied by the Providence limestone of Allegheny age. The section from the top of No. 11 coal to the base of No. 12 is as follows:

BASE: Top of Coal, No. 11	Ft.	In.
(1) Clay, soft, fine-grained, dark red, sour taste; gypsum crystals.....	1	2
(2) Limestone, massive; crystalline at base, but becoming very shaly in top 6 inches (Providence).....	1	6
(3) Clay, soft, tobacco brown; many gypsum crystals, and limestone fragments; decidedly sour taste.....	0	6
(4) Clay, soft, light gray; probably fire clay; sour taste.....	1	2
TOP: Base of Coal No. 12		

It is believed that this occurrence of gypsum is a result of the common reaction of acid waters from a coal seam with a limestone. The acid water resulted from the oxidation of iron pyrite contained in the coal.

In the case under discussion, both Coal No. 11, below, and Coal No. 12, above, are potential sources of supply.

It will be noted that there is a clay zone immediately underlying, and overlying, the limestone bed. Within these two zones the well developed selenite crystals are found in profusion.

The position of the two clay zones above and below the limestone suggests two possibilities concerning the movement of the acid solution. First, it is possible that there have been two sources of acid: that is, it may have been derived not only from the upper coal but also from the No. 11 seam below. In the former case, any acid which formed in Coal No. 12 probably percolated downward into the upper layers of the Providence Limestone, with the resultant formation of selenite crystals. In the latter case, the sulfuric acid formed in Coal No. 11 was brought in contact with the overlying limestone through capillary action.

The second hypothesis is that all of the sulfuric acid responsible for the leached zones in which the selenite occurs has been formed in the upper seam, or Coal No. 12, and has seeped downward; a portion of the acid reacting with the top part of the limestone, and the remainder of the solution descending through open joint planes of the bed until reaching the bottom layers. In this case, the lower coal bed, No. 11, probably acted as a relatively impervious stratum, causing a concentration of the acid solution on its upper surface.

The writer is inclined to believe that the latter alternative fits the observed conditions, because of the open character of the joint fractures which permit free passage of the acid waters downward to the base of the limestone bed. Additional support for this hypothesis lies in the fact that concentrations of crystals seemed to occur at the intersection of joint planes with the leached zones above, and below, the limestone horizon. Also, the joint planes are filled with the same brown ferruginous clay as that associated with the upper and lower surface of the limestone, thus indicating additional acid leaching as it passed downward through the bed.

Some suggestions have been made that the crystals may have formed at the same time as the limestone. The fact that many of the crystals contain inclusions of clay, and the occurrence of two zones of the selenite above and below the limestone, seems to prove their secondary origin conclusively. Furthermore, the clay residue possesses a distinctly sour taste which probably indicates a considerable acid content now present in the rock. In fact, it is entirely possible that the selenite is being formed today.

PROCEEDINGS OF SOCIETIES
MINERALOGICAL SOCIETY OF GREAT BRITAIN AND IRELAND

Meeting held June 24, 1937

The following papers were read.

(1) *Wollastonite solid-solutions from Scawt Hill, Co. Antrim.* By Prof. C. E. TILLEY
Wollastonites with distinctive optics comparable with those of synthetic solid solutions in the system CaO-FeO-SiO_2 are recorded from hybrid rocks (wollastonite-bearing dolerites) of the Scawt Hill contact zone.

(2) *Notes on silicate synthesis with a laboratory gas furnace.* By Dr. A. T. DOLLAR.

Comparisons are made between methods of synthesizing silicates, in a high-temperature research laboratory, with (a) a Fletcher gas-blast crucible furnace, and (b) a vertical electric crucible furnace of platinum resistance type, having approximately the same dimensions. Utility of the gas furnace is discussed in relation to the bulk, homogeneity, rate of production and cost of silicate melts which it yields. Technical details are illustrated by reference to the preparation of diopside glass.

(3) *Some new pyroxenes included in the system clinoenstatite, clinohypersthene, diopside, hedenbergite.* By Mr. W. A. DEER and Mr. L. R. WAGER.

Four pyroxenes from the Skaergaard Halvoen intrusion, Kangerdlugsuak, East Greenland, have been analysed. They occur in a series varying from a hypersthene olivine-gabbro at the base to a fayalite-quartz-gabbro at the top of the crystallisation-differentiated gabbro complex. The pyroxene, as well as the rocks in which they occur, show a remarkable increase in the percentage of ferrous iron. The pyroxenes are all low in sesquioxides and may be regarded as approximating in composition to members of the clinoenstatite-clinohypersthene-diopside-hedenbergite system. Two of the pyroxenes are very rich in the clinohypersthene molecule and extend the field of previously investigated natural augites. The most ferri-ferrous type is almost a pure member of Bowen's (1935) hedenbergite-clinohypersthene solid solution series, and contains nearly 70 per cent FeSiO_3 .

(4) *A new micro-pyknometric method for the specific gravity of heavy solids: with a note on the accuracy of specific gravity determinations.* By Mr. F. A. BANNISTER and Dr. MAX H. HEY.

A straight silica tube of $\frac{1}{2}$ mm. bore, closed at one end, serves as a variable volume pyknometer. The accuracy of the method may reach about 0.5 per cent with 5 to 30 milligrams of material.

(5) *An occurrence of corundum at Fraserburgh, Aberdeenshire.* By Mr. ROBERT WALLS.

Corundum is found at several places on the coast at Fraserburgh as small, usually irregular, grains embedded in muscovite, in a biotite-feldspar rock. The beds at Fraserburgh are metamorphosed sediments of the andalusite-schist-facies. It is suggested that the corundum bearing rocks were derived from andalusite-schist (into which they pass) by the desilicating action of alkali-aluminate emanations from neighbouring pegmatites. Analogous cases in France and Australia are mentioned.

(6) *A review of the data of the Mg-Fe clinopyroxenes.* By Mr. N. F. M. HENRY.

The identification of clinoenstatite and clinohypersthene from igneous rocks in all the reports that have been examined rests on insufficient evidence, mainly on inclined extinctions alone. The available data for the series is summarised.

(7) *Fourteenth list of new mineral names.* By Dr. L. J. SPENCER.

(8) *Paragenesis of kyanite-amphibolites.* By Prof. C. E. TILLEY, with chemical analyses by Mr. H. C. G. VINCENT.

The genesis of the assemblage hornblende-kyanite in metamorphic rocks is briefly discussed. Special reference is made to the problems offered by kyanite-bearing amphibolites and eclogites of the Glenelg area (Scotland).